

NEW CHEMOSENSOR SYSTEMS OF THE BENZO- [de]ISOQUINOLINE-1,3-DIONE SERIES

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New derivatives of the benzo[de]isoquinoline-1,3-dione system containing an amino group were synthesized by the reaction of 2-benzyl-6-bromobenzo[de]isoquinoline-1,3-dione with ethylenediamine and hydrazine. Further functionalization of the free amino groups leads to imines, amines, thioureas, and hydrazones. Some compounds exhibit high chemosensor selectivity in the determination of anions.

Keywords: aminoethylamines, azomethines, benzo[de]isoquinoline-1,3-diones (1,8-naphthalimides), hydrazines, hydrazones, chemosensor activity, fluorescence.

Earlier we reported that *N,N'*-bis(anthracen-9-ylmethyl)alkanediamines and their derivatives exhibit the properties of highly effective fluorescent chemosensors for various cations: H⁺, Zn²⁺, Cd²⁺, Hg²⁺ etc. [1-5]. The principal mechanism of the action of these sensor systems is the PET (photoinduced electron transfer) effect [6-9].

Variation of the fluorophore structure present in such systems leads not only to a change in the sensor properties, but also to a change of the working effect. Thus, PCT (photoinduced charge transfer) becomes dominant in cases where derivatives of 1,8-naphthalimide are used as signal fragment [6, 9].

Naphthalimide systems are widely used as effective fluorescent and colorimetric sensors for cations and anions [10-14]. Of special interest are compounds that contain a free amino group capable of further transformation, particularly with the introduction of additional complex-forming fragments and/or fluorophores. It is, thus, possible to vary the effectiveness, selectivity, and other parameters of the sensors.

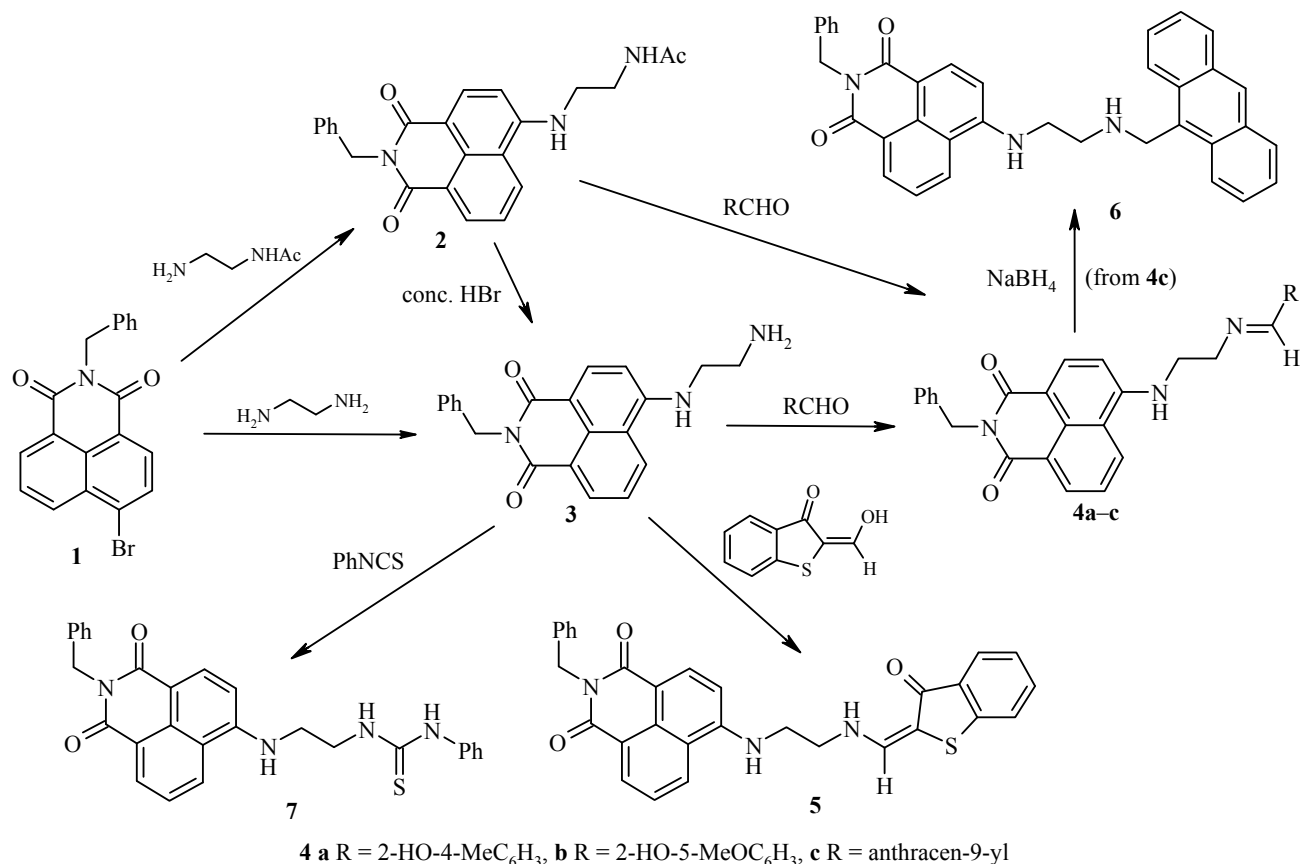
Potential synthons for ionochromic sensors are benzo[de]isoquinoline-1,3-diones containing aminoalkyl or hydrazine fragments [12, 13]. In order to investigate further chemosensors with polyamine receptors we synthesized compounds **2-10**. The diamine **3** was obtained by the reaction of the bromine derivative **1** with ethylenediamine [12] or *N*-(2-aminoethyl)acetamide, followed by acidic hydrolysis of the amide **2**. The ¹H NMR spectra of the derivatives **2** and **3** contain characteristic signals for the protons of the CH₂CH₂ fragment and also for the amine and amide groups.

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The reaction of aromatic aldehydes and 2-hydroxymethylenebenzo[*b*]thiophen-3-one with the amine **3** gave a series of imines **4a-c** and also the derivative **5**. The reactions are accompanied by disappearance of the signal for the NH₂ group protons in the ¹H NMR spectra and by the appearance of signals for additional aromatic and CH protons. Compounds **4a-c** were also obtained by the reaction of the corresponding aldehydes with the acetamide **2** upon heating in 1-butanol.



Reduction of the azomethine **4c** in a mixture of DMF and ethanol gave the diamine system **6** with a double fluorophore. In the ¹H NMR spectrum of this compound, the proton signals for the alkyl chain CH₂ groups show a substantial upfield shift of 0.4 and 1.07 ppm, compared to the analogous signals of the derivative **4c**, and there is also a signal for the protons of the CH₂-Ant fragment at 4.70 ppm.

The amine **3** was modified by the introduction of a thiourea fragment. Irrespective of the reaction conditions and of the amount of phenyl isothiocyanate used it was only possible to introduce one thiourea molecule. This is probably due to steric hindrances.

Investigation of the luminescence spectral and chemosensor properties of the obtained derivatives shows that, contrary to previously described derivatives of similar type [10-13], their chemosensor properties are nonspecific. Thus, the addition of the divalent metal acetates (Zn²⁺, Cd²⁺, Cu²⁺, Co²⁺, Ni²⁺, Pb²⁺, Hg²⁺) to acetonitrile solutions of the naphthalimides **4a-c**, **5-7** leads to a 30-300% change in the fluorescence intensity, but poor selectivity in the determination of ions is observed.

When compound **1** was heated with hydrazine hydrate, the previously unknown amine **8** was isolated as the main reaction product instead of the expected 2-benzyl-6-hydrazinobenzo[*de*]isoquinoline-1,3-dione [14].

The structure of the amine **8** is confirmed by disappearance of the signals for the benzyl substituent protons and by the appearance of signals for the NH₂ and NH₂NH fragment protons in the ¹H NMR spectra. Compound **8** was also obtained by heating 6-bromobenzo[*de*]isochromene-1,3-dione with hydrazine hydrate.

In the reaction of the obtained naphthalimide **8** with anthracene-9-carbaldehyde, irrespective of the reaction conditions and the amount of the aldehyde used in the reaction, only the hydrazine fragment enters into condensation with the formation of the hydrazone **9**, and the signal for the NH₂ group protons remains in the ¹H NMR spectrum. In contrast to this, 2-hydroxynaphthalene-1-carbaldehyde adds to the naphthalimide **8** at both NH₂ groups with the formation of the diimine **10**.

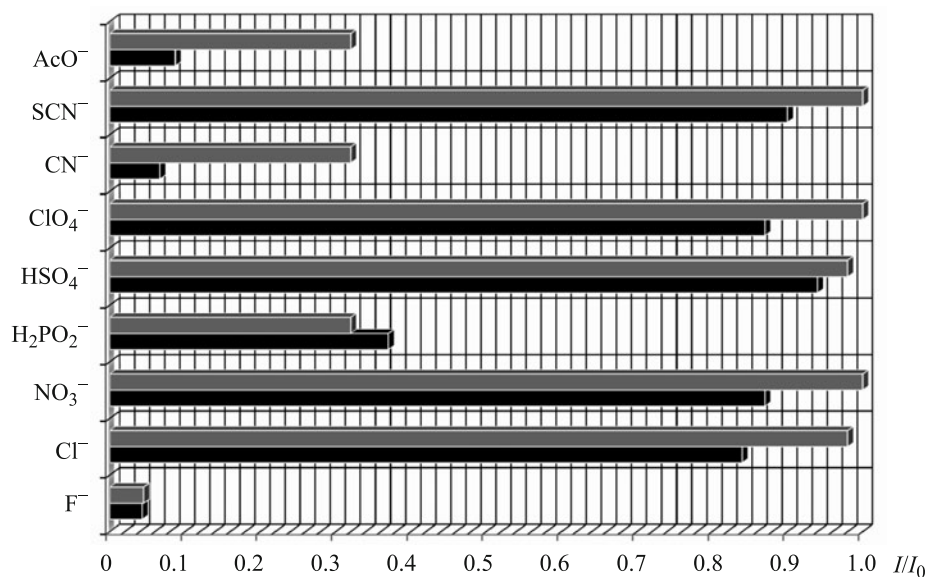
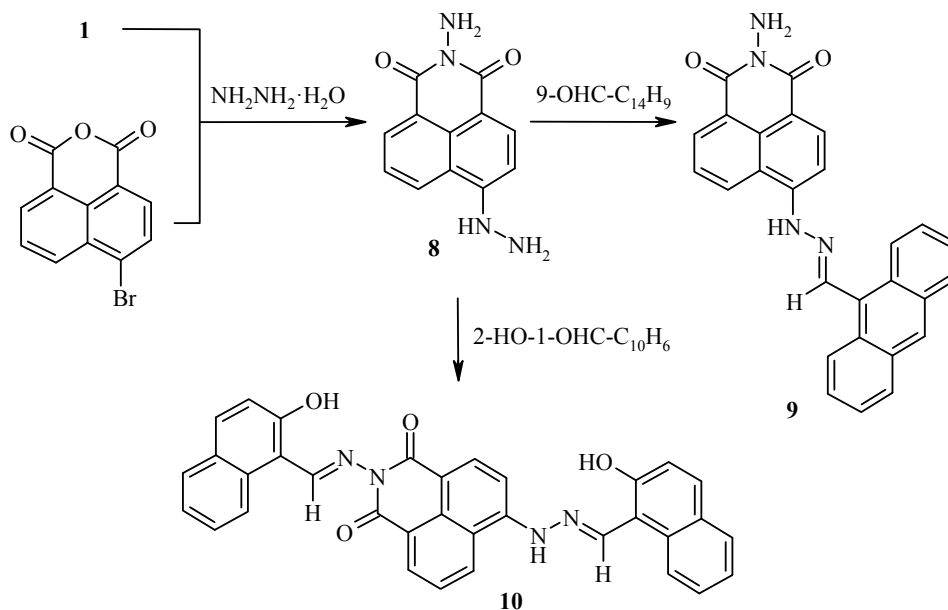


Fig. 1. The variation of the fluorescence intensity (I/I_0) of compounds **9** (■) and **10** (■) (c 5.0×10^{-6} M) in MeCN upon the addition of salts NBu_4^+A^- (c 2.5×10^{-5} M).

According to the data from fluorescence spectra, the imines **9** and **10**, like compounds **3-7**, exhibit a weak chemosensor activity with respect to cations. In both cases the most significant changes in luminescence intensity are caused by Zn²⁺ (buildup) and Cu²⁺ (quenching).

The ionochromic activity with respect to F^- , Cl^- , CN^- , SCN^- , NO_3^- , $H_2PO_4^-$, ClO_4^- , HSO_4^- , and AcO^- ions was investigated for these compounds (Fig. 1). Addition of the corresponding tetrabutylammonium salts to acetonitrile solutions of compounds **9** and **10** leads to quenching of the fluorescence, and the interaction is most selective with F^- (23 times (**9**) and 22 times (**10**)), CN^- (15 times (**9**) and three times (**10**)), and AcO^- anions (11 times (**9**) and 3 times (**10**)). The presented data indicate that the selectivity for fluoride ions increases in the transition from the imine **9** to the diimine **10**. At the same time, the interaction with F^- anions gives rise to a substantial hypsochromic shift by 55 and 63 nm, respectively (a change in the fluorescence color).

Thus, a series of new benzo[*de*]isoquinoline-1,3-dione derivatives containing additional complex-forming fragments has been synthesized. The investigations of the chemosensor activity show that 2-[(2-hydroxynaphthalen-1-ylmethylene)amino]-6-[*N'*-(2-hydroxynaphthalen-1-ylmethylene)hydrazino]benzo[*de*]isoquinoline-1,3-dione is a highly selective reagent for F^- ions.

EXPERIMENTAL

The IR spectra were recorded on a Varian Excalibur 3100 FT-IR instrument. The 1H NMR spectra were recorded in DMSO- d_6 on a Varian Unity 300 spectrometer (300 MHz) with the residual signals of the solvent as internal standard (δ 2.50 ppm). The electronic absorption spectra were recorded on a Varian Cary 100 spectrophotometer, and the luminescence spectra were recorded on a Varian Cary Eclipse fluorescence spectrometer. Element analysis was performed on a Euro Vector EA-3000 elemental analyzer. The melting points were determined in glass capillaries on a PTP (M) instrument. The reaction progress and the purity of the obtained compounds were monitored by TLC (Silufol UV-254 plates, eluent $CHCl_3$, development with iodine vapor in a humidity chamber).

***N*-[2-(2-Benzyl-1,3-dioxo-2,3-dihydro-1*H*-benzo[*de*]isoquinolin-6-ylamino)ethyl]acetamide (2).** 2-Benzyl-6-bromobenzo[*de*]isoquinoline-1,3-dione (**1**) (1.83 g, 5 mmol) was dissolved in EtOH (50 ml), *N*-(2-aminoethyl)acetamide (1.0 g, 10 mmol) was added, and the mixture was refluxed for 20 h. The solvent was evaporated on a rotary evaporator, and the residue was recrystallized from a 1:2 mixture of benzene and petroleum ether. Yield 1.20 (60%). Light-beige fibrous crystals, mp 256-257°C. IR spectrum, ν , cm^{-1} : 3300, 3220 (NH), 1700, 1665, 1650 (C=O), 1600, 1545 (C=C), 1370 (C–N–C). 1H NMR spectrum, δ , ppm (*J*, Hz): 1.90 (3H, s, $COCH_3$); 3.80 (1H, br. s, NHAr); 3.40-3.70 (4H, m, $(CH_2)_2$); 5.15 (2H, s, CH_2Ph); 6.90 (H, d, *J* = 8.2, H Ar); 7.10-7.84 (6H, m, H Ar); 8.41-8.67 (3H, m, H Ar); 10.40 (1H, br. s, CONH). Found, %: C 71.25; H 5.52; N 10.79. $C_{23}H_{21}N_3O_3$. Calculated, %: C 71.30; H 5.46; N 10.85.

6-(2-Aminoethylamino)-2-benzyl-1*H*-benzo[*de*]isoquinoline-1,3(2*H*)-dione (3). A. A 46% HBr solution (2 ml) was added to a solution of the acetamide **2** (0.77 g, 2 mmol) in 2-PrOH (20 ml). The mixture was heated for 2 h. It was then cooled, diluted with H_2O (20 ml), and neutralized by adding Na_2CO_3 ; the amine **3** was extracted with $CHCl_3$ (3×20 ml). The extract was evaporated, and the residue was crystallized from a 1:1 mixture of benzene and petroleum ether. Yield 0.52 g (75%). Light-cream colored powder, mp 239-240°C. IR spectrum, ν , cm^{-1} : 3320, 3150 (NH_2 , NH), 1705, 1655 (C=O), 1590, 1530 (C=C), 1380 (C–N–C). 1H NMR spectrum, δ , ppm (*J*, Hz): 3.10-3.22 (3H, m, NH_2 , NH); 3.23 (2H, t, *J* = 7.2, $CH_2CH_2NH_2$); 3.60 (2H, t, *J* = 7.2, $CH_2CH_2NH_2$); 5.17 (2H, s, CH_2Ph); 6.75 (1H, d, *J* = 8.2, H Ar); 7.15-7.90 (6H, m, H Ar); 8.46-8.75 (3H, m, H Ar). Found, %: C 72.95; H 5.61; N 12.22. $C_{21}H_{19}N_3O_2$. Calculated, %: C 73.03; H 5.54; N 12.17.

B. The amine **3** was obtained through a direct reaction of the dione **1** with ethylenediamine under conditions analogous to the production of compound **2**. Yield 62%; mp 238-239°C. The spectral characteristics agree with those of the compound produced by method A.

Compounds 4a-c, 5 (General Method). A. A mixture of the amine **3** (0.69 g, 2 mmol) with the corresponding aldehyde (2 mmol) in EtOH (10 ml) (for the preparation of compounds **4a-c**) or with 2-hydroxymethylenebenzo[*b*]thiophen-3-one (0.36 g, 2 mmol) in MeCN (10 ml) (for the preparation of compound **5**)

was heated for 2 h. The mixture was cooled, and the precipitate was filtered off and crystallized from a suitable solvent.

B. The azomethines **4a-c** were obtained by refluxing the acetamide **2** (2 mmol) for 10 h with an equimolar amount of the corresponding aldehyde in 1-BuOH (20 ml). Yields 70-75%.

2-Benzyl-6-{2-[(2-hydroxy-4-methylbenzylidene)amino]ethylamino}-1H-benzo[de]isoquinoline-1,3(2H)-dione (4a). Yield 0.75 g (81%). Yellow crystals, mp 203-204°C (PhMe). IR spectrum, ν , cm^{-1} : 3200 (NH, OH), 1695, 1655 (C=O), 1640 (C=N), 1610, 1550 (C=C), 1375 (C–N–C). ^1H NMR spectrum, δ , ppm (J , Hz): 2.28 (3H, s, CH_3); 3.10-3.25 (2H, m, ArNHCH_2); 3.60-3.80 (2H, m, $\text{CH}_2\text{N}=\text{C}$); 5.21 (2H, s, CH_2Ph); 6.80 (1H, d, $J = 8.4$, H Ar); 7.10-7.70 (7H, m, H Ar); 8.00 (1H, s, NHAr); 8.20-8.52 (5H, m, $\text{N}=\text{CH}$, H Ar); 8.92 (H, d, $J = 8.4$, H Ar). Found, %: C 75.20; H 5.52; N 8.99. $\text{C}_{29}\text{H}_{25}\text{N}_3\text{O}_3$. Calculated, %: C 75.14; H 5.44; N 9.07.

2-Benzyl-6-{2-[(2-hydroxy-5-methoxybenzylidene)amino]ethylamino}-1H-benzo[de]isoquinoline-1,3(2H)-dione (4b). Yield 0.74 g (77%). Orange fibrous crystals, mp 232-233°C (PhMe). IR spectrum, ν , cm^{-1} : 3150 (NH, OH), 1700, 1660 (C=O), 1645 (C=N), 1600, 1560 (C=C), 1375 (C–N–C). ^1H NMR spectrum, δ , ppm (J , Hz): 3.60-4.00 (7H, m, OCH_3 , $(\text{CH}_2)_2$); 5.20 (2H, s, CH_2Ph); 6.63-6.90 (4H, m, H Ar); 7.10-7.42 (5H, m, H Ar); 7.60 (1H, t, $J = 7.6$, H Ar); 7.82 (1H, br. s, ArNH); 8.28 (1H, d, $J = 8.0$, H Ar); 8.34-8.50 (2H, m, $\text{N}=\text{CH}$, H Ar); 8.61 (1H, d, $J = 8.0$, H Ar); 12.50 (1H, s, OH). Found, %: C 72.70; H 5.30; N 8.70. $\text{C}_{29}\text{H}_{25}\text{N}_3\text{O}_4$. Calculated, %: C 72.64; H 5.25; N 8.76.

6-{2-[(Anthracen-9-ylmethylene)amino]ethylamino}-2-benzyl-1H-benzo[de]isoquinoline-1,3(2H)-dione (4c). Yield 0.92 g (86%). Fine yellow crystals, mp 190-191°C (decomp., 1-BuOH). IR spectrum, ν , cm^{-1} : 3320, 3210 (NH), 1690, 1670 (C=O), 1640 (C=N), 1620, 1550 (C=C), 1375 (C–N–C). ^1H NMR spectrum, δ , ppm (J , Hz): 3.88-4.00 (2H, m, ArNHCH_2); 4.27 (2H, t, $J = 7.2$, $\text{CH}_2\text{N}=\text{C}$); 5.22 (2H, s, CH_2Ph); 6.91 (1H, d, $J = 8.2$, H Ar); 7.10-7.50 (9H, m, H Ar); 7.60 (1H, t, $J = 7.8$, H Ar); 7.88-8.08 (3H, m, H Ar, ArNH); 8.25 (1H, d, $J = 8.6$, H Ar); 8.33-8.54 (5H, m, H Ar); 9.53 (1H, s, $\text{N}=\text{CH}$). Found, %: C 81.11; H 5.06; N 7.94. $\text{C}_{36}\text{H}_{27}\text{N}_3\text{O}_2$. Calculated, %: C 81.03; H 5.10; N 7.87.

2-Benzyl-6-{2-[(3-oxo-3H-benzo[b]thiophen-2-ylidenemethyl)amino]ethylamino}-2-benzyl-1H-benzo[de]isoquinoline-1,3(2H)-dione (5). Yield 0.69 g (68%). Red-brown amorphous crystals, mp 188-189°C (toluene). IR spectrum, ν , cm^{-1} : 3300, 3180 (NH), 1700, 1660 (C=O), 1600, 1545 (C=C), 1380 (C–N–C). ^1H NMR spectrum, δ , ppm (J , Hz): 3.55-3.82 (4H, m, $(\text{CH}_2)_2$); 5.20 (2H, s, CH_2Ph); 6.83 (1H, t, $J = 8.2$, H Ar); 7.10-7.84 (11H, m, H Ar); 8.02 (1H, br. s, ArNH); 8.22-8.50 (2H, m, H Ar); 8.63 (1H, t, $J = 8.2$, $\text{NHCH}=\text{}$); 10.60 (1H, br. s, $\text{NHCH}=\text{}$). Found, %: C 71.35; H 4.64; N 8.23; S 6.28. $\text{C}_{30}\text{H}_{23}\text{N}_3\text{O}_3\text{S}$. Calculated, %: C 71.27; H 4.59; N 8.31; S 6.34.

6-{2-[(Anthracen-9-ylmethyl)amino]ethylamino}-2-benzyl-1H-benzo[de]isoquinoline-1,3(2H)-dione (6). NaBH_4 (0.23 g, 6 mmol) was added over 5 min with stirring and heating (40-50°C) to a suspension of the azo-methine **4c** (1.10 g, 2 mmol) in EtOH (50 ml). The solution was stirred for 2 h and diluted with H_2O (100 ml), and the excess of the borohydride was neutralized by the addition of dilute AcOH. The mixture was cooled, and the precipitate was filtered off, dried, and crystallized from 1-BuOH. Yield 0.95 g (88%). Light-orange needle shaped crystals, mp 176-177°C (decomp.). IR spectrum, ν , cm^{-1} : 3250, 3180 (NH), 1710, 1665 (C=O), 1600, 1550 (C=C), 1370 (C–N–C). ^1H NMR spectrum, δ , ppm (J , Hz): 3.20 (2H, t, $J = 7.4$, $\text{CH}_2\text{CH}_2\text{NHCH}_2$); 3.48-3.61 (2H, m, ArNHCH_2); 4.70 (2H, s, NHCH_2Ar); 5.22 (2H, s, CH_2Ph); 6.84 (1H, d, $J = 8.4$, H Ar); 7.10-7.60 (11H, m, H Ar); 7.90-8.05 (2H, m, H Ar); 8.24 (1H, d, $J = 8.4$, H Ar); 8.35-8.50 (5H, m, H Ar, ArNH). Found, %: C 80.80; H 5.40; N 7.90. $\text{C}_{36}\text{H}_{29}\text{N}_3\text{O}_2$. Calculated, %: C 80.72; H 5.46; N 7.84.

1-[2-(2-Benzyl-1,3-dioxo-2,3-dihydro-1H-benzo[de]isoquinolin-6-ylamino)ethyl]-3-phenylthiourea (7). A mixture of the amine **3** (0.7 g, 2 mmol) and phenyl isothiocyanate (0.6 ml, 5 mmol) in benzene (10 ml) was refluxed for 5 h. The mixture was cooled, and the precipitate was filtered off, then crystallized from 1-BuOH. Yield 0.8 g (83%). Light-yellow coarsely crystalline powder, mp 297-298°C. IR spectrum, ν , cm^{-1} : 3375 (NH), 1705, 1660 (C=O), 1584, 1537 (C=C), 1375 (C–N–C). ^1H NMR spectrum, δ , ppm (J , Hz): 3.52-3.67 (2H, m, ArNHCH_2); 3.81-3.97 (2H, m, CH_2NHCS); 5.20 (2H, s, CH_2Ph); 6.88 (1H, d, $J = 8.2$, H Ar); 7.00-7.43 (10H, m, H Ar); 7.60 (1H, t, $J = 7.6$, H Ar); 7.72-7.93 (2H, m, ArNH, NHCS); 8.27 (1H, d, $J = 8.2$,

H Ar); 8.43 (1H, d, $J = 8.0$, H Ar); 8.64 (1H, d, $J = 8.2$, H Ar); 9.60 (1H, s, NHPh). Found, %: C 70.05; H 4.97; N 11.52; S 6.60. $C_{28}H_{24}N_4O_2S$. Calculated, %: C 69.98; H 5.03; N 11.66; S 6.67.

2-Amino-6-hydrazino-1H-benzo[de]isoquinoline-1,3(2H)-dione (8). A. 85% Hydrazine hydrate (5 ml) was added to a solution of 4-bromonaphthalic anhydride (1.1 g, 4 mmol) in butyl cellosolve (40 ml). The reaction mixture was refluxed for 2 h and cooled. The precipitate was filtered off and washed thoroughly with MeOH. The product was crystallized from a 1:1 mixture of 1-BuOH and DMF and dried at 110-115°C. Yield 0.84 g (87%). Dark-yellow finely crystalline powder, mp >300°C (decomp.). IR spectrum, ν , cm^{-1} : 3300, 3250 (NH₂, NH), 1705, 1670 (C=O), 1700, 1655 (C=O), 1600, 1555 (C=C), 1375 (C-N). ¹H NMR spectrum, δ , ppm (J , Hz): 5.38 (2H, s, NNH₂); 7.20-7.35 (2H, m, H Ar); 7.48-7.58 (2H, m, H Ar); 7.77-7.90 (1H, m, H Ar); 8.48-8.72 (3H, m, NHNH₂). Found, %: C 59.43; H 4.22; N 23.20. $C_{12}H_{10}N_4O_2$. Calculated, %: C 59.50; H 4.16; N 23.13.

B. In the reaction of compound **1** with hydrazine hydrate under conditions similar to those described above, the hydrazine **8** was obtained. Yield 78%; mp >300°C (decomp.). The spectral characteristics agree with those of the compound obtained by method A.

2-Amino-6-[N'-(anthracen-9-ylmethylene)hydrazino]-1H-benzo[de]isoquinoline-1,3(2H)-dione (9). The hydrazine **8** (0.48 g, 2 mmol) was dissolved in 1-BuOH (10 ml), anthracene-9-carbaldehyde (0.41 g, 2 mmol) was added, and the mixture was refluxed for 2 h. The mixture was cooled, and the precipitate was filtered off, washed with MeOH, and crystallized from a 4:1 mixture of 1-BuOH and DMF. Yield 0.75 g (87%). Dark-orange crystals, mp >330°C (decomp.). IR spectrum, ν , cm^{-1} : 3210 (NH₂, NH), 1690, 1655 (C=O), 1645 (C=N), 1590, 1500 (C=C), 1375 (C-N). ¹H NMR spectrum, δ , ppm (J , Hz): 5.69 (2H, s, NH₂), 7.42-8.90 (14H, m, H Ar); 9.67 (1H, s, N=CH); 11.62 (1H, s, NH). Found, %: C 75.40; H 4.15; N 12.96. $C_{27}H_{18}N_4O_2$. Calculated, %: C 75.34; H 4.21; N 13.02.

2-[(2-Hydroxynaphthalen-1-ylmethylene)amino]-6-[N'-(2-hydroxynaphthalen-1-ylmethylene)hydrazino]-1H-benzo[de]isoquinoline-1,3(2H)-dione (10). This compound was obtained similarly to compound **9** in the reaction of the hydrazine **8** (0.48 g, 2 mmol) and 2-hydroxynaphthalene-1-carbaldehyde (0.86 g, 5 mmol). Yield 0.79 g (72%). Dark red-brown finely crystalline powder, mp >220°C (decomp., 1-BuOH-DMF, 3:1). IR spectrum, ν , cm^{-1} : 3300, 3220 (NH), 1695, 1660 (C=O), 1645, 1640 (C=N), 1600, 1500 (C=C), 1375 (C-N). ¹H NMR spectrum, δ , ppm (J , Hz): 7.10-8.97 (17H, m, H Ar); 9.65 (1H, s, CH=N); 9.93 (1H, s, NHN=CH); 11.43-11.64 (2H, m, OH, NH); 12.80 (1H, s, OH). Found, %: C 74.25; H 3.96; N 10.24. $C_{34}H_{22}N_4O_4$. Calculated, %: C 74.17; H 4.03; N 10.18.

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