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Photochromic Benzo[*g*]indolyl Fulgimide with Modulated Fluorescence

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Abstract—A new photochromic fulgimide, (*E*)-1-(anthracen-9-ylmethyl)-3-[1-(5-methoxy-2-methyl-1-phenyl-1*H*-benzo[*g*]indol-3-yl)ethylidene]-4-(propan-2-ylidene)pyrrolidine-2,5-dione, has been synthesized and found to exhibit fluorescence. The structure of this compound and intermediate fulgenates and amidofulgenic and fulgenic acids has been determined by electronic and vibrational spectroscopy, ^{1}H and ^{13}C NMR, and mass spectrometry. The amidofulgenic and fulgenic acids are capable of undergoing photoinduced reversible *Z*/*E* isomerization with respect to the C=C bond without subsequent cyclization, whereas fulgenates are converted to the corresponding cyclic structures. The new fluorescent fulgimide is transformed into the colored non-fluorescing cyclic isomer under UV irradiation. The reverse ring opening under visible light irradiation restores the fluorescence properties, which makes this compound a molecular fluorescence switch.

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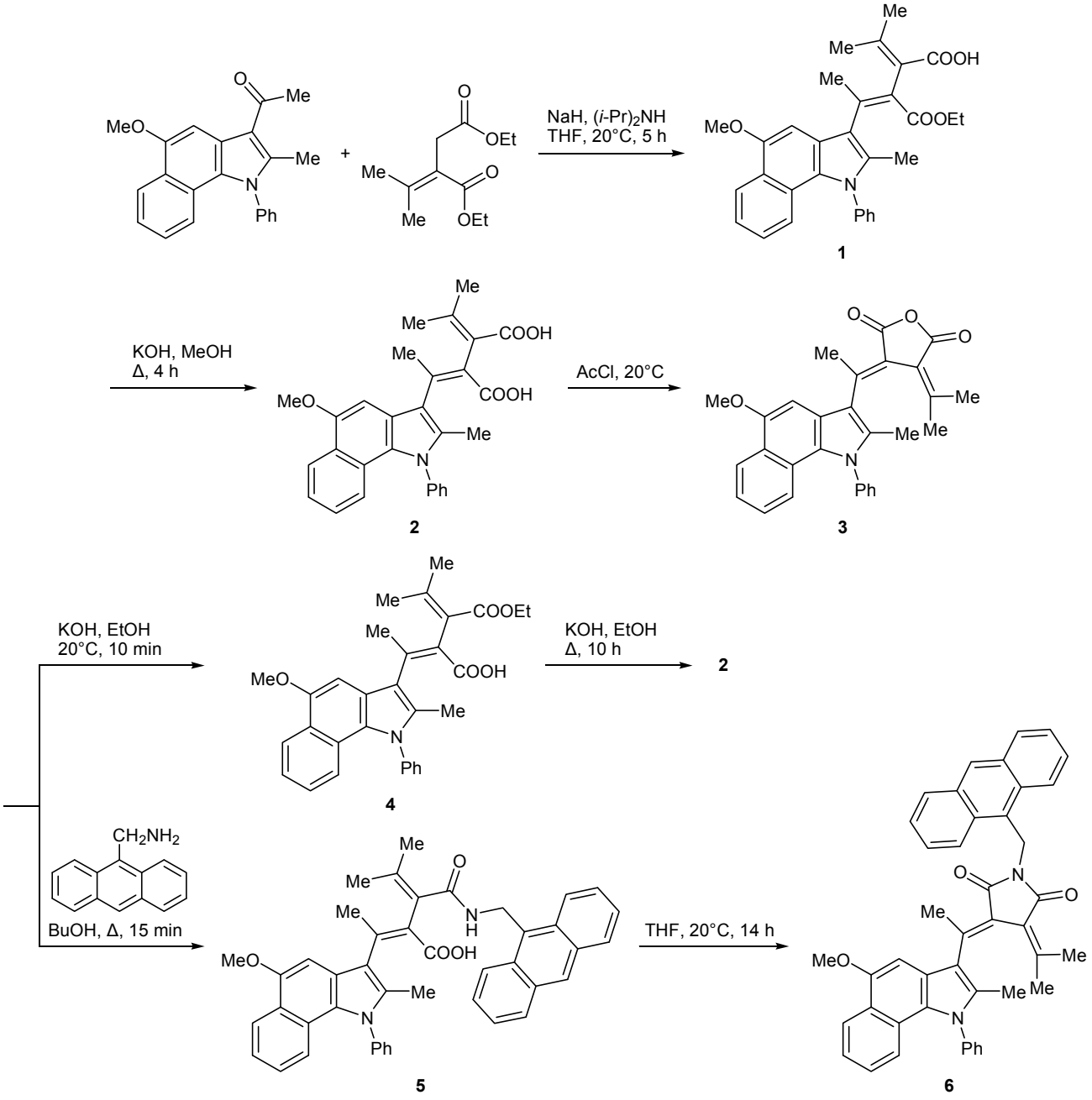
Heterocyclic fulgides and fulgimides are the most studied photochromic compounds [1–5]. Their irradiation with UV light induces rearrangement of the hexatriene structure to isomeric cyclohexadiene. Due to high thermal stability and resistance to photodegradation of colored cyclic forms, fulgides and fulgimides are considered to be efficient molecular switches and promising materials for three-dimensional optical memory systems [6–11]. If one or both isomeric forms involved in the photorearrangement of fulgides and fulgimides exhibit fluorescence properties, such compounds offer particular advantages in nondestructive readout of optical information. We previously studied electronic absorption and luminescence spectra of a series of fulgides containing oxazole, naphthofuran, benzothiophene, indole, and benzoindole substituents [12–16]. With the goal of obtaining new molecular switches, in the present work we have synthesized a new photochromic benzo[*g*]indolylfulgimide and studied its electronic absorption and luminescence spectra and photochemical behavior, as well as the corresponding properties of its synthetic precursors, derivatives of amidofulgenic and fulgenic acids.

Fulgenate **1** was synthesized by the Stobbe reaction of 3-acetyl-5-methoxy-2-methyl-1-phenyl-1*H*-benzo[*g*]indole and diethyl 2-isopropylidenesuccinate in THF in the presence of sodium hydride and diisopropylamine according to the procedure described in [14]. Hydrolysis of **1** gave fulgenic acid **2** which was converted to fulgide **3** by treatment with acetyl chloride [14]. Fulgenate **4** was prepared by dissolution of **3** in 10% ethanolic potassium hydroxide, and the subsequent hydrolysis of **4** also afforded acid **2**. By heating fulgide **3** with (anthracen-9-yl)methanamine in butan-1-ol for a short time we obtained amidofulgenic acid **5** which was subjected to intramolecular cyclization to fulgimide **6** by the action of carbonyldiimidazole in THF (Scheme 1).

The structure of **1**–**6** was confirmed by IR, ^{1}H and ^{13}C NMR, and mass spectra. The IR spectra of **1**, **2**, and **4** contained absorption bands in the region 1666–1707 cm^{-1} due to stretching vibrations of the ester and acid carbonyl groups, and stretching vibrations of O–H bonds involved in strong hydrogen bonds were observed as broadened bands in the region 2300–3600 cm^{-1}. Amidofulgenic acid **5** showed in the IR

Scheme 1.



spectrum an absorption band at 1721 cm^{-1}, which is typical of amide carbonyl stretching vibrations, and the broad band in the region 2300–3250 cm^{-1} was assigned to the strongly H-bonded O–H group. Stretching vibrations of the NH group appeared at 3372 cm^{-1}. The two carbonyl groups in fulgide **3** and fulgimide **6** gave rise to IR absorption bands in the region 1691–1750 cm^{-1}.

Compounds **1**–**6** displayed in the ^1H NMR spectra signals of protons in the methyl groups of the ethylidene and benzoindole fragments at δ 0.73–2.89 ppm and a more downfield signal of the 5-methoxy group (δ 3.70–4.00 ppm). In keeping with published data [17], the position of the methyl proton signals indicated *E* configuration of the exocyclic C=C double bond in both **3** and **6**. In addition, the ^1H NMR spectra of **5** and **6** contained signals of aromatic protons in the anthracene fragment and *AB* multiplets at δ 5.45–5.84 ppm from diastereotopic protons of the bridging methylene group. These data suggest that molecules **5** and **6** are chiral with a high racemization barrier,

Parameters of the electronic absorption and fluorescence spectra of compounds **1**–**6** in acetonitrile at 293 K[a]

Compound no.	Open isomer **O**		Cyclic isomer **C**	
	λ_{max}(abs.), nm ($\varepsilon\times10^{-3}$, L mol^{-1} cm^{-1})	λ_{max}(fluor.), nm (I_{fl}, rel. units)	λ_{max}(abs.), nm (A_{max})	λ_{max}(fluor.), nm (I_{fl}, rel. units)
1	284 (19.5), 324 (6.2)	384 (217)	477 (0.06)	–
2	284 (22.5), 331 (7.6)	387 (520)	–	
3 [14]	373 (10.80)	–	643 (0.19)	791 (242)
4	285 (20.8), 324 (6.3)	384 (209)	476 (0.06)	–
5	347 (15.4), 365 (16.9), 387 (15.3)	421 (205)	–	
6	348 (16.7), 367 (17.3), 386 (13.6)	424 (193)	612 (0.37)	–

[a] λ_{max} stands for absorption and fluorescence maxima, I_{fl} is the fluorescence intensity, and A_{max} is the optical density at the absorption maximum of photoinduced isomer **C** in the photostationary state.

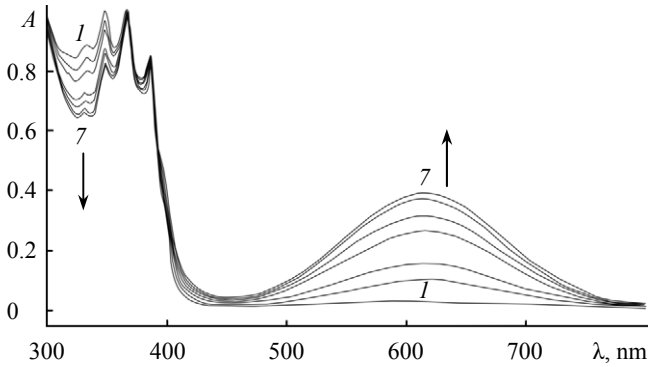
which is related to nonplanar arrangement of substituents on the double bond in the benzoindolylethylidene fragment for steric reasons. The ^{1}H NMR spectrum of **2** characteristically showed a broadened downfield two-proton signal of the hydroxy groups, and the spectrum of **5** displayed NH and OH signals. In the ^{1}H NMR spectra of **1** and **4** we observed a broadened downfield OH signal and two multiplets from protons of the ester ethyl group. The *ABX*$_3$ multiplet of the OCH_2CH_3 group was not broadened, indicating diastereotopicity of the methylene protons and chirality of molecules **1** and **4** with a high barrier to racemization (cf. the data for **5** and **6**). The ^{13}C NMR spectra of **1**–**6** lacked signals of quaternary carbon atoms typical of the dihydrobenzocarbazole isomers, which confirmed their acyclic structure.

The electronic absorption spectra of fulgenates **1** and **4** and fulgenic acid **2** showed maxima at λ 284–285 and 324–331 nm with molar absorption coefficients of 19460–22480 and 6200–7600 L mol^{-1} cm^{-1}, respectively (see table). Amidofulgenic acid **5** and fulgimide **6** were characterized by broad fine-structure bands in the region λ 347–387 nm, which are typical of anthracene chromophore [18] (see table and figure). Unlike non-fluorescing fulgide **3**, fulgimide **6** in acetonitrile solution showed fluorescence at λ 424 nm (λ_{excit} 369 nm).

The photochemical behavior of compounds **1**, **2**, and **4**–**6** was studied in acetonitrile solution. Irradiation of fulgenic acid **2** at λ 313 or 365 nm induced its *Z*/*E* isomerization without subsequent cyclization. Irradiation of fulgenates **1** and **4** and fulgimide **6** with a mercury lamp at λ 313 and 365 nm, respectively, resulted in photocoloration due to appearance of new absorption bands at λ 476–477 (**1**, **4**) and 612 nm (**6**) whose intensity increased in the course of irradiation, whereas the intensity of the original absorption band decreased (see figure). This pattern is typical of photocyclization **O**→**C** [1, 2] (Scheme 2) until photostationary state is reached.

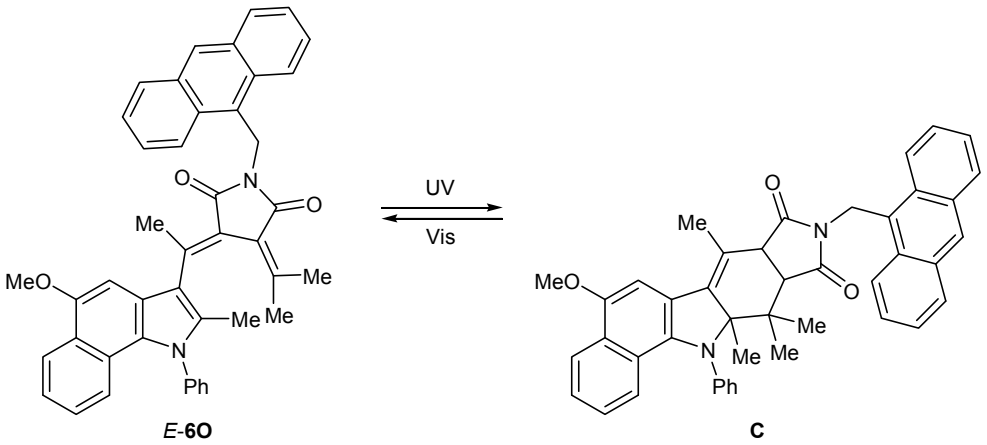
The formation of photostationary state involves significant overlap of absorption bands arising from the $S_0\rightarrow S_1$ transition in initial form **O** and $S_0\rightarrow S_2$ transition in photoinduced isomer **C** [5, 13, 14]. The fluorescence intensity of open isomer **6O** decreases as the intensity of the new longwave band at λ 612 nm increases without change of the position of the emission maximum; unlike fulgide **3C**, cyclic isomer **6C** exhibits no fluorescence properties (see table).

Cyclic form **6C** absorbs at shorter wavelengths than does **3C** (see table) and is less thermally stable than **3C**; the latter is stable for at least 72 h [14]. Isomer **6C** at room temperature in the dark undergoes gradual decoloration with a rate constant $k_{\mathbf{C}\rightarrow\mathbf{O}}$ of 1.74×10^{-5} s^{-1}. Irradiation of a blue solution of **6C** at λ 436 nm also induces decoloration due to the reverse reaction



Electronic absorption spectra of fulgimide **6** in acetonitrile ($c = 6.0\times10^{-5}$ M) (*1*) before and after irradiation at λ 365 nm for (*2*) 30, (*3*) 60, (*4*) 180, (*5*) 300, (*6*) 600, and (*7*) 900 s.

Scheme 2.



E-**6O** **C**

6C → *E*-6O and almost completely restores the initial spectral parameters. Photocontrolled transformations **6O ↔ 6C** can be repeated many times without appreciable reduction of molar absorption coefficients at the absorption maxima.

In summary, we have synthesized a new photochromic benzo[*g*]indolylfulgimide possessing switchable fluorescence properties via Stobbe reaction. Irradiation of this compound gives rise to non-fluorescing cyclic isomer. Its synthetic precursors, fulgenic and amidofulgenic acid, are capable of undergoing only reversible *Z*/*E* photoisomerization with respect to the C=C bond without subsequent cyclization. Like the fulgimide, the corresponding fulgenates are converted into cyclic isomers on photoexcitation.

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were recorded on Varian Unity 300 (300 MHz) and Bruker Avance-600 (150 MHz) spectrometers from solution in $CDCl_3$ or DMSO-d_6 using the residual proton signal of the solvent as reference ($CHCl_3$, δ 7.26 ppm; DMSO-d_5, δ 2.49 ppm). The electronic absorption spectra were measured on a Varian Cary 100 spectrophotometer, and the luminescence spectra were recorded on a Varian Cary Eclipse spectrofluorimeter. The IR spectra were taken on a Varian Excalibur 3100 FT-IR instrument equipped with a ZnSe ATR accessory. The mass spectra (electron impact, 70 eV) were obtained on a Shimadzu GCMS-QP2010SE instrument with direct sample admission into the ion source. The melting ponts were determined in glass capillaries using a PTP (M) melting point apparatus.

3-(Ethoxycarbonyl)-4-(5-methoxy-2-methyl-1-phenyl-1*H*-benzo[*g*]indol-3-yl)-2-(propan-2-ylidene)-pent-3-enoic acid (1) was synthesized according to the procedure described in [14]. Yield 61%, mp 212–214°C (from toluene); published data [14]: mp 222–223°C. IR spectrum, ν, cm^{-1}: 3200–2400 (OH), 1707, 1682. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.73 t (3H, CH_2**Me**, J = 7.1 Hz), 2.01 s (3H, Me), 2.13 s (3H, Me), 2.19 s (3H, Me), 2.26 s (3H, Me), 3.73–3.94 m (2H, CH_2), 4.00 s (3H, OMe), 6.91 s (1H, H_{arom}), 7.00–7.62 m (8H, H_{arom}), 8.29 d (1H, H_{arom}, J = 8.4 Hz), 11.64 br.s (1H, OH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 170.9, 169.4, 150.5, 150.3, 144.4, 140.6, 132.2, 129.9, 129.8, 129.2, 129.1, 129.0, 128.8, 125.6, 125.5, 125.3, 123.8, 123.3, 123.2, 122.8, 122.6, 120.1, 116.8, 96.4, 61.1, 55.7, 23.3, 22.6, 22.2, 13.5, 11.8. Mass spectrum, m/z (I_{rel}, %): 497 (100) $[M]^+$, 498 (34) $[M + H]^+$, 482 (17), 453 (13), 392 (13), 287 (15), 272 (16).

2-[1-(5-Methoxy-2-methyl-1-phenyl-1*H*-benzo[*g*]indol-3-yl)ethylidene]-3-(propan-2-ylidene)-butanedioic acid (2) was synthesized according to the procedure described in [14]. Yield 94%, mp 248–249°C (from toluene); published data [14]: mp 245–246°C. IR spectrum, ν, cm^{-1}: 3400–2300 (OH), 1687, 1666. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.94 s (3H, Me), 2.05 s (3H, Me), 2.06 s (3H, Me), 2.19 s (3H, Me), 3.92 s (3H, OMe), 6.92–7.70 m (9H, H_{arom}), 8.16 d (1H, H_{arom}, J = 8.4 Hz), 11.88 br.s (2H, OH). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 168.3, 167.9, 149.3, 147.3, 140.0, 139.6, 132.3, 131.6, 130.1, 130.0, 128.9, 128.8, 128.6, 126.5, 125.3, 124.7, 123.1, 122.7, 122.6, 122.3, 122.2, 119.4, 117.1, 97.1, 55.4, 23.4, 22.4, 21.7, 11.6. Mass spectrum, m/z (I_{rel}, %): 469 (100) $[M]^+$, 470 (32) $[M + H]^+$, 454 (19), 451 (25), 364 (19), 287 (19), 272 (19).

(*E*)-3-[1-(5-Methoxy-2-methyl-1-phenyl-1*H*-benzo[*g*]indol-3-yl)ethylidene]-4-(propan-2-ylidene)-

tetrahydrofuran-2,5-dione (3) was synthesized according to the procedure described in [14]. Yield 95%, mp 182–184°C (from MeCN) [14]. IR spectrum, ν, cm^{-1}: 1800, 1750. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.13 s (3H, Me), 2.07 s (3H, Me), 2.20 s (3H, Me), 2.89 s (3H, Me), 3.99 s (3H, OMe), 6.68 s (1H, H_{arom}), 6.96–7.68 m (8H, H_{arom}), 8.33 d (1H, H_{arom}, J = 7.7 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 164.1, 163.7, 153.8, 151.0, 149.1, 139.8, 134.6, 130.3, 130.2, 129.4, 128.9, 128.5, 126.04, 126.0, 124.1, 123.4, 123.3, 122.7, 121.5, 121.2, 120.2, 120.0, 119.0, 96.7, 55.8, 26.1, 23.8, 22.7, 12.7. Mass spectrum, *m*/*z* (I_{rel}, %): 451 (100) $[M]^+$, 452 (33) $[M + H]^+$, 436 (17), 408 (11), 406 (12), 364 (22), 287 (14), 77 (13).

3-(Ethoxycarbonyl)-2-[1-(5-methoxy-2-methyl-1-phenyl-1*H*-benzo[*g*]indol-3-yl)ethylidene]-4-methylpent-3-enoic acid (4). Fulgide **3**, 100 mg (0.22 mmol), was dissolved under stirring in 3 mL of a 10% solution of potassium hydroxide in ethanol at room temperature. The colorless solution was poured into 50 mL of water, and the fine solid was filtered off through a folded filter. The filtrate was acidified to pH 1 with 10% aqueous HCl, and the precipitate of **4** was filtered off, washed with water, dried in air, and recrystallized from acetonitrile. Yield 102 mg (94%), mp 206–207°C. IR spectrum, ν, cm^{-1}: 3600–2400 (OH), 1707, 1682. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.29 t (3H, CH_2**Me**, J = 7.1 Hz), 1.72 s (3H, Me), 1.80 s (3H, Me), 1.87 s (3H, Me), 2.58 s (3H, Me), 3.95 s (3H, OMe), 4.19–4.34 m (2H, ***AB***X_3, OCH_2), 6.82 s (1H, H_{arom}), 6.97 d (1H, H_{arom}, J = 8.5 Hz), 7.03–7.60 m (9H, H_{arom}, OH), 8.24 d (1H, H_{arom}, J = 8.2 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 171.3, 168.4, 151.0, 150.2, 146.9, 140.6, 133.2, 130.0, 129.7, 129.5, 129.3, 128.9, 128.7, 125.9, 125.8, 125.4, 123.7, 123.0, 122.8, 122.5, 122.3, 120.1, 117.1, 97.0, 60.5, 55.8, 24.9, 23.0, 22.5, 14.3, 11.9. Mass spectrum, *m*/*z* (I_{rel}, %): 498 (34) $[M + H]^+$, 497 (100) $[M]^+$, 482 (17), 451 (28), 392 (13), 364 (14), 287 (17), 272 (14). Found, %: C 74.69; H 6.17; N 2.99. $C_{31}H_{31}NO_5$. Calculated, %: C 74.83; H 6.28; N 2.81. *M* 497.59.

3-[(Anthracen-9-ylmethyl)carbamoyl]-2-[1-(5-methoxy-2-methyl-1-phenyl-1*H*-benzo[*g*]indol-3-yl)ethylidene]-4-methylpent-3-enoic acid (5). Fulgide **3**, 75.8 mg (0.14 mmol), was added to a solution of 31.1 mg (0.15 mmol) of (anthracen-9-yl)methanamine in 3 mL of butan-1-ol, and the mixture was refluxed for 15 min. The light yellow crystals were filtered off, dried in air, and used further without additional purification. Yield 78 mg (71%), mp 279–280°C. IR spectrum, ν, cm^{-1}: 3372 (NH), 2300–3250 (OH), 1721. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 1.57 s (3H, Me), 1.58 s (3H, Me), 1.79 s (3H, Me), 2.21 s (3H, Me), 3.88 s (3H, OMe), 5.45 m (2H, ***ABX***, CH_2), 6.78–8.70 m (21H, H_{arom}, OH, NH). Mass spectrum, *m*/*z* (I_{rel}, %): 658 (5) $[M]^+$, 640 (9), 452 (32), 451 (100), 364 (35), 287 (25), 272 (25), 207 (60), 206 (47), 191 (92),189 (41), 179 (37), 178 (74), 176 (39), 77 (84). Found, %: C 80.03; H 5.93; N 4.09. $C_{44}H_{38}N_2O_4$. Calculated, %: C 80.22; H 5.81; N 4.25. *M* 658.80.

(*E*)-1-(Anthracen-9-ylmethyl)-3-[1-(5-methoxy-2-methyl-1-phenyl-1*H*-benzo[*g*]indol-3-yl)-ethylidene]-4-(propan-2-ylidene)pyrrolidine-2,5-dione (6). *N*,*N*′-Carbonyldiimidazole, 45.4 mg (0.28 mmol), was added with stirring to a suspension of 78 mg (0.12 mmol) of amido acid **5** in 5 mL of anhydrous THF. The mixture gradually became homogeneous and turned light yellow. It was stirred for 14 h at room temperature, the solvent was distilled off under reduced pressure, and the residue was treated with 10 mL of water and extracted with ethyl acetate. The organic extract was evaporated, and the residue was purified by silica gel column chromatography using petroleum ether–ethyl acetate (3:1) as eluent. The product was additionally recrystallized from methanol. Yield 32 mg (42%), colorless crystals, mp 263–264°C. IR spectrum, ν, cm^{-1}: 1741, 1691. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.02 s (3H, Me), 1.95 s (3H, Me), 2.08 s (3H, Me), 2.79 s (3H, Me), 3.70 s (3H, OMe), 5.84 q (2H, *AB*, CH_2), 6.52 s (1H, H_{arom}), 6.94 d (1H, H_{arom}, J = 8.4 Hz), 7.10–8.70 m (17H, H_{arom}). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 169.2, 168.6, 150.5, 148.0, 142.8, 140.2, 133.9, 131.5, 131.1, 130.1, 130.0, 129.1, 129.0, 128.7, 128.4, 126.3, 126.2, 126.0, 125.7.2, 125.6, 125.2, 124.9, 124.87, 124.0, 123.90, 123.87, 123.2, 122.9, 122.6, 121.6, 120.1, 96.8, 55.5, 35.3, 25.9, 22.9, 22.0, 12.5. Mass spectrum, *m*/*z* (I_{rel}, %): 641 (23) $[M + H]^+$, 640 (56) $[M]^+$, 462 (14), 364 (16), 312 (19), 225 (17), 192 (12), 191 (100), 189 (24). Found, %: C 82.58; H 5.53; N 4.19. $C_{44}H_{36}N_2O_3$. Calculated, %: C 82.47; H 5.66; N 4.37. *M* 640.78.

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