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Chemistry of Heterocyclic Compounds **2021**, *57*(6), 691–694

SHORT COMMUNICATIONS

Oxa-[3+3] annulation of 1*H*-benzo[*f*]chromene-2-carbaldehydes and 2-naphthols: synthesis of 7a*H*,15*H*-benzo[*f*]benzo[5,6]chromeno[2,3-*b*]chromenes

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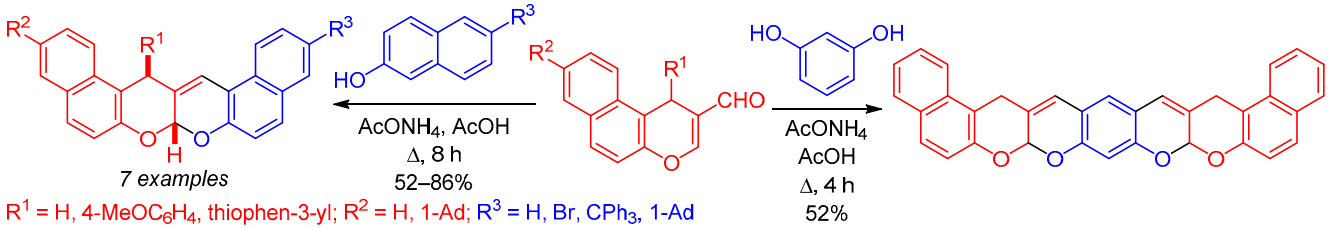


A method for the preparation of polycondensed chromeno[2,3-*b*]chromenes was developed based on the formal [3+3] cycloaddition reaction between 1*H*-benzo[*f*]chromene-2-carbaldehydes and 2-naphthols. In the case of resorcinol, the bisannulation product formed with the participation of both hydroxy groups was isolated.

Keywords: 1*H*-benzo[*f*]chromene-2-carbaldehydes, chromeno[2,3-*b*]chromenes, 2-naphthols, electrocyclization, formal [3+3] cycloaddition.

Interest in the development of methods for the preparation of chromeno[2,3-*b*]chromenes is primarily due to the presence of this structural fragment in the composition of many compounds of plant origin possessing antitumor,[1] antiplasmodic,[2] neuroprotective,[3] anti-inflammatory,[4] and other types of biological activity. The α-glucosidase inhibitor yunanensin A isolated from plants of the genus *Morus*,[5] the natural antioxidant mongolicin B,[6] the phosphodiesterase I inhibitor mesozygin A,[7] and mulberrofuran F,[8] exhibiting antibacterial activity against vancomycin-resistant enterococci, can be cited as examples (Fig. 1).

One of the most commonly used methods for the preparation of chromeno[2,3-*b*]chromenes is the [4+2] cycloaddition with the participation of *in situ* generated *o*-quinone methides[9] and various dienophiles, including α-chloroacrylonitrile,[10] 4*H*-chromenes,[11] 1,1-bis(morpholino)-ethylene,[12] together with the dimerization of 2*H*-chromenes.[13,14] The synthesis of chromeno[2,3-*b*]chromene derivatives based on salicylic aldehydes and ketones[15] or chromones[16] is known. In addition, two examples of the preparation of chromeno[2,3-*b*]chromenes as a result of the Claisen rearrangement of 2-(aryloxymethyl)-1*H*-benzo[*f*]-chromenes, which, in turn, were synthesized from 1*H*-benzo[*f*]chromene-2-carbaldehyde in three steps, have been published.[17] In this case, the rearrangement itself

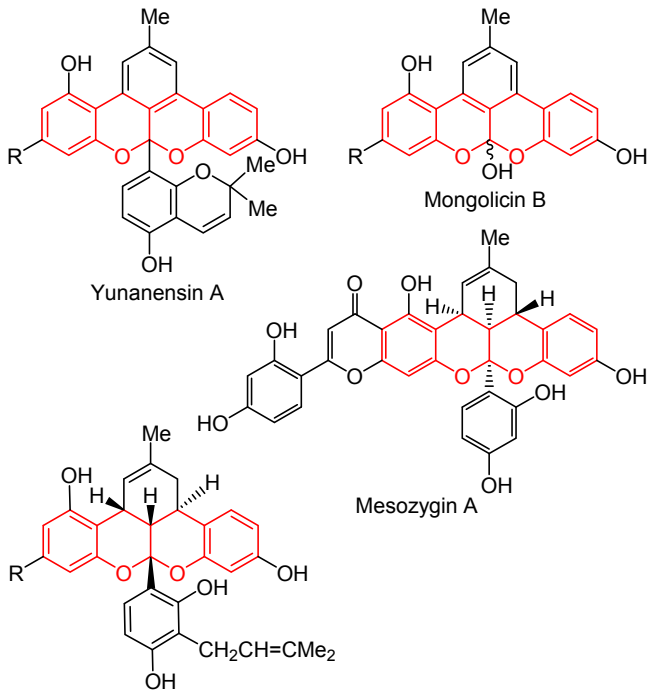
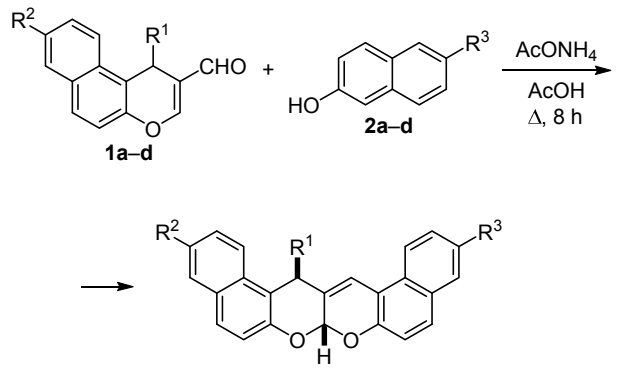


Figure 1. Biologically active chromeno[2,3-*b*]chromenes of natural origin.

proceeded under rather harsh conditions with prolonged heating under reflux in *N*,*N*-diethylaniline.

In this work, we propose a one-step method for the preparation of polycondensed 7a*H*,15*H*-benzo[*f*]benzo[5,6]-chromeno[2,3-*b*]chromenes **3a**–**g** from 1*H*-benzo[*f*]chromene-2-carbaldehydes **1a**–**d** and 2-naphthols **2a**–**d** (Scheme 1). The reactions were carried out by heating an equimolar mixture of starting compounds **1** and **2** in AcOH under reflux for 8 h in the presence of $AcONH_4$. The yields of 7a*H*,15*H*-benzo[*f*]benzo[5,6]chromeno[2,3-*b*]chromenes **3a**–**g** varied from 52 to 86%. By studying the reaction of 1*H*-benzo[*f*]chromene-2-carbaldehyde **1a** with unsubstituted 2-naphthol **2a**, it was found that the best yield of product **3a** (70%) is achieved when using 2 equiv of $AcONH_4$. In the presence of 1.5 equiv $AcONH_4$, the reaction time increased to 20 h and the yield of chromeno-[2,3-*b*]chromene **3a** was 62%. The reaction did not proceed in the absence of $AcONH_4$. The use of *p*-TsOH instead of $AcONH_4$ resulted in resinification of the reaction mixture. It should be noted that in the case of 1-substituted aldehydes **1c**,**d** the reaction proceeded diastereoselectively with the formation of *trans*-isomers **3f**,**g**.

Scheme 1

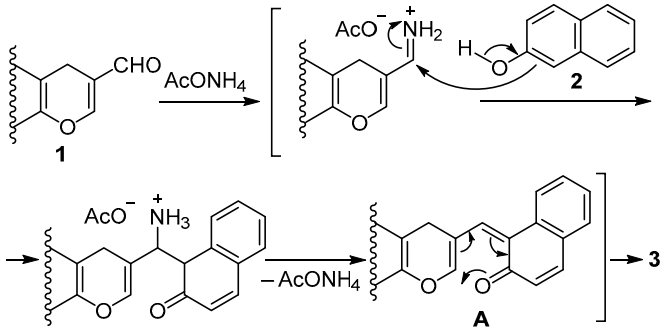


3a $R^1 = R^2 = R^3 = H$ (70%)
b $R^1 = R^2 = H$, $R^3 = Br$ (67%)
c $R^1 = R^2 = H$, $R^3 = CPh_3$ (52%)
d $R^1 = R^2 = H$, R^3 = 1-Ad (68%)
e $R^1 = R^3 = H$, R^2 = 1-Ad (75%)
f R^1 = 4-$MeOC_6H_4$, $R^2 = R^3 = H$ (75%)
g R^1 = thiophen-3-yl, $R^2 = R^3 = H$ (86%)

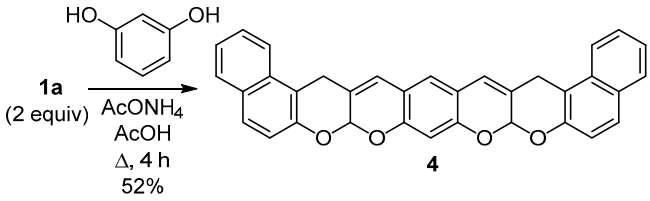
Apparently, the presence of $AcONH_4$ is necessary for the activation of the carbonyl group of the aldehyde by transforming it into a more electrophilic iminium salt. Subsequent nucleophilic attack by naphthol and deamination lead to the generation of highly reactive 1,2-naphthoquinone 1-methide **A** which undergoes disrotatory oxa-6π electrocyclization (Scheme 2).

The reaction of aldehyde **1a** with resorcinol (in a ratio of 2:1) in the presence of $AcONH_4$ (2 equiv) led to the symmetric product **4** which was isolated in 52% yield (Scheme 3). Subjecting phloroglucinol to this transformation led to a complex mixture of unidentified compounds, whereas the less nucleophilic hydroquinone, pyrocatechol, and monohydric phenols (4-*tert*-butyl-, 2-methoxy-, and 3,5-dimethylphenols) were unreactive.

Scheme 2



Scheme 3



In the ^1H NMR spectra of compounds **3a**–**e**, the diastereotopic methylene protons appear as two separate doublets in the intervals of 4.02–4.06 and 4.17–4.19 ppm with a coupling constant of 17.9–18.1 Hz. The spectra of compounds **3f**,**g** are characterized by the signal of the proton 15-CH in the form of a singlet at 5.63–5.73 ppm. The signal of the acetal proton 7a-CH in the spectra of chromenochromenes **3a**–**g** is found in the 6.65–6.79 ppm region. In the ^{13}C NMR spectra, the methylene (products **3a**–**e**, **4**) and acetal (products **3a**–**g**, **4**) carbon atoms resonate in the ranges of 30.6–31.4 and 93.3–96.1 ppm, respectively. Based on the established configuration of the previously obtained thiochromeno[3',4':5,6]pyrano[2,3-*b*]-chromen-6-ones[18] as well as the absence of cross peaks corresponding to the interaction of protons 7a-CH and 15-CH in the NOESY spectra of compounds **3f**,**g**, the respective protons were assigned the *trans* arrangement in relation to each other.

To conclude, we developed a method for the preparation of 7a*H*,15*H*-benzo[*f*]benzo[5,6]chromeno[2,3-*b*]chromenes based on a heterodomino reaction involving electrophilic substitution in 2-naphthols and oxa-6π electrocyclization with the participation of *in situ* generated 1,2-naphthoquinone 1-methides.

Experimental

^1H and ^{13}C NMR (400 and 100 MHz, respectively) as well as DEPT-135 spectra were registered on a JEOL JNM-ECX400 spectrometer in DMSO-d_6 (compounds **3**,**b**,**c**,**f**,**g**, and **4**) or $CDCl_3$ (compounds **3a**,**d**,**e**); the residual solvent signals (DMSO-d_6: 2.50 ppm for ^1H nuclei and 39.5 ppm for ^{13}C nuclei; $CDCl_3$: 7.26 ppm for ^1H nuclei and 77.0 ppm for ^{13}C nuclei) served as internal standard. Elemental analysis was performed on a Euro Vector EA-3000 CHNS-analyzer. Melting points were determined by the capillary method on an SRS OptiMelt MPA100 apparatus. Monitoring of the reaction progress and assessment of the purity of synthesized compounds were done by TLC on

Merck Silica gel 60 F_{254} plates, eluent $CHCl_3$, visualization with UV light or by iodine stain.

The starting 1*H*-benzo[*f*]chromene-2-carbaldehydes **1a**–**d** were obtained by published methods.[18,19]

Synthesis of 7a*H*,15*H*-benzo[*f*]benzo[5,6]chromeno-[2,3-*b*]chromenes 3a–g (General method). A mixture of 1*H*-benzo[*f*]chromene-2-carbaldehyde **1a**–**d** (1 mmol), 2-naphthol **2a**–**d** (1 mmol), $AcONH_4$ (155 mg, 2 mmol), and AcOH (5 ml) was heated under reflux for 8 h. After cooling the reaction mixture to room temperature, the formed precipitate was filtered off, washed with ice-cold MeOH (2 ml), and recrystallized from AcOH.

7a*H*,15*H*-Benzo[*f*]benzo[5,6]chromeno[2,3-*b*]chromene (3a). Yield 235 mg (70%), colorless crystals, mp 210–211°C (mp 194°C[17]). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.03 (1H, d, *J* = 18.1, CH_2); 4.18 (1H, d, *J* = 18.1, CH_2); 6.67 (1H, s, 7a-CH); 7.12 (1H, d, *J* = 8.9, H Ar); 7.30 (1H, d, *J* = 8.7, H Ar); 7.36–7.43 (2H, m, H Ar); 7.44 (1H, s, 16-CH); 7.49–7.57 (2H, m, H Ar); 7.67 (1H, d, *J* = 8.9, H Ar); 7.73 (1H, d, *J* = 8.9, H Ar); 7.78–7.84 (3H, m, H Ar); 8.05 (1H, d, *J* = 8.5, H Ar). ^{13}C NMR spectrum, δ, ppm: 31.4 (CH_2); 96.0 (7a-CH); 111.2; 114.5; 115.7 (CH); 117.4 (CH); 119.2 (CH); 121.0 (CH); 122.0 (CH); 124.1 (CH); 124.2 (CH); 125.7; 126.9 (3CH); 128.7 (2CH); 128.8 (CH); 129.4; 129.5; 129.6 (CH); 130.0; 132.3; 148.2; 150.9. Found, %: C 85.60; H 4.84. $C_{24}H_{16}O_2$. Calculated, %: C 85.69; H 4.79.

3-Bromo-7a*H*,15*H*-benzo[*f*]benzo[5,6]chromeno[2,3-*b*]-chromene (3b). Yield 280 mg (67%), colorless crystals, mp 233–234°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.06 (1H, d, *J* = 17.9, CH_2); 4.19 (1H, d, *J* = 17.9, CH_2); 6.79 (1H, s, 7a-CH); 7.05 (1H, d, *J* = 8.7, H Ar); 7.35 (1H, d, *J* = 8.5, H Ar); 7.38–7.42 (1H, m, H Ar); 7.55–7.59 (1H, m, H Ar); 7.64 (1H, d, *J* = 8.5, H Ar); 7.70–7.75 (2H, m, 16-CH, H Ar); 7.83–7.89 (3H, m, H Ar); 8.16–8.21 (2H, m, H Ar). ^{13}C NMR spectrum, δ, ppm: 31.1 (CH_2); 95.5 (7a-CH); 112.0; 115.2 (CH); 115.4; 117.9; 118.8 (CH); 119.1 (CH); 122.8 (CH); 124.5 (CH); 124.6 (CH); 127.5 (CH); 127.9; 128.1; 128.9 (2CH); 129.2 (CH); 129.4; 130.3 (CH); 130.8 (CH); 131.3; 132.3; 148.2; 150.7. Found, %: C 69.45; H 3.61. $C_{24}H_{15}BrO_2$. Calculated, %: C 69.41; H 3.64.

3-Trityl-7a*H*,15*H*-benzo[*f*]benzo[5,6]chromeno[2,3-*b*]-chromene (3c). Yield 300 mg (52%), light-yellow crystals, mp 185–186°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.03 (1H, d, *J* = 18.1, CH_2); 4.18 (1H, d, *J* = 18.1, CH_2); 6.76 (1H, s, 7a-CH); 7.05 (1H, d, *J* = 9.0, H Ar); 7.15–7.31 (17H, m, H Ar); 7.39 (1H, t, *J* = 7.4, H Ar); 7.55 (1H, t, *J* = 7.1, H Ar); 7.61 (1H, s, 16-CH); 7.65 (1H, d, *J* = 1.8, H-4); 7.68 (1H, d, *J* = 8.9, H Ar); 7.72 (1H, d, *J* = 9.0, H Ar); 7.83 (1H, d, *J* = 7.8, H Ar); 7.86 (1H, d, *J* = 8.5, H Ar); 8.09 (1H, d, *J* = 9.0, H Ar). ^{13}C NMR spectrum, δ, ppm: 31.2 (CH_2); 64.9 (CPh_3); 95.6 (7a-CH); 111.6; 115.4 (CH); 115.5; 117.6 (CH); 119.2 (CH); 121.3 (CH); 122.8 (CH); 124.5 (CH); 126.6 (3CH Ph); 127.5 (2CH); 127.6; 128.4 (6CH Ph); 128.8 (CH); 128.9 (2CH); 129.3; 129.4; 130.3 (CH); 131.1 (6CH Ph); 131.5 (CH); 132.4; 142.9; 146.7 (3C Ph); 148.1; 150.8. Found, %: C 89.31; H 5.19. $C_{43}H_{30}O_2$. Calculated, %: C 89.25; H 5.23.

3-(Adamantan-1-yl)-7a*H*,15*H*-benzo[*f*]benzo[5,6]chromeno[2,3-*b*]chromene (3d). Yield 320 mg (68%), light-yellow crystals, mp 185–186°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.76–1.84 (6H, m, CH_2 Ad); 2.01 (6H, br. s, CH_2 Ad); 2.14 (3H, br. s, CH Ad); 4.03 (1H, d, *J* = 17.9, CH_2); 4.17 (1H, d, *J* = 17.9, CH_2); 6.66 (1H, s, 7a-CH); 7.12 (1H, d, *J* = 8.9, H Ar); 7.27 (1H, d, *J* = 8.9, H Ar); 7.38–7.43 (2H, m, 16-CH, H Ar); 7.53–7.57 (1H, m, H Ar); 7.61 (1H, dd, *J* = 8.9, *J* = 1.6, H Ar); 7.65–7.68 (2H, m, H Ar); 7.71 (1H, d, *J* = 8.9, H Ar); 7.79 (1H, d, *J* = 8.2, H Ar); 7.82 (1H, d, *J* = 8.5, H Ar); 8.00 (1H, d, *J* = 8.9, H Ar). ^{13}C NMR spectrum, δ, ppm: 29.0 (3CH Ad); 31.4 (CH_2); 36.2 (C Ad); 36.9 (3CH_2 Ad); 43.2 (3CH_2 Ad); 96.1 (7a-CH); 111.0; 114.6; 115.8 (CH); 117.1 (CH); 119.2 (CH); 120.8 (CH); 122.0 (CH); 123.7 (CH); 124.0 (CH); 125.1 (CH); 125.4; 126.9 (CH); 127.6; 128.7 (2CH); 129.5; 129.6 (CH); 130.0; 132.3; 147.1; 147.8; 150.9. Found, %: C 86.82; H 6.35. $C_{34}H_{30}O_2$. Calculated, %: C 86.78; H 6.43.

12-(Adamantan-1-yl)-7a*H*,15*H*-benzo[*f*]benzo[5,6]-chromeno[2,3-*b*]chromene (3e). Yield 353 mg (75%), light-yellow crystals, mp 209–210°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.77–1.85 (6H, m, CH_2 Ad); 2.02 (6H, br. s, CH_2 Ad); 2.14 (3H, br. s, CH Ad); 4.02 (1H, d, *J* = 18.1, CH_2); 4.17 (1H, d, *J* = 18.1, CH_2); 6.65 (1H, s, 7a-CH); 7.09 (1H, d, *J* = 8.9, H Ar); 7.30 (1H, d, *J* = 8.9, H Ar); 7.36–7.40 (1H, m, H Ar); 7.43 (1H, s, 16-CH); 7.49–7.53 (1H, m, H Ar); 7.63–7.67 (3H, m, H Ar); 7.73 (1H, d, *J* = 8.9, H Ar); 7.77–7.80 (2H, m, H Ar); 8.05 (1H, d, *J* = 8.7, H Ar). ^{13}C NMR spectrum, δ, ppm: 29.0 (3CH Ad); 31.4 (CH_2); 36.2 (C Ad); 36.9 (3CH_2 Ad); 43.2 (3CH_2 Ad); 96.0 (7a-CH); 111.3; 114.3; 115.5 (CH); 117.4 (CH); 119.0 (CH); 121.1 (CH); 121.8 (CH); 123.8 (CH); 124.1 (CH); 125.1 (CH); 125.9; 126.9 (CH); 128.7 (2CH); 129.4; 129.5 (CH, C); 130.0; 130.4; 147.0; 148.2; 150.4. Found, %: C 86.85; H 6.37. $C_{34}H_{30}O_2$. Calculated, %: C 86.78; H 6.43.

***trans*-15-(4-Methoxyphenyl)-7a*H*,15*H*-benzo[*f*]benzo-[5,6]chromeno[2,3-*b*]chromene (3f)**. Yield 330 mg (75%), colorless crystals, mp 243–244°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 3.65 (3H, s, OCH_3); 5.63 (1H, s, 15-CH); 6.55 (1H, s, 7a-CH); 6.84 (2H, d, *J* = 8.7, H Ar); 7.15 (1H, d, *J* = 8.9, H Ar); 7.20 (2H, d, *J* = 8.7, H Ar); 7.24 (1H, d, *J* = 8.9, H Ar); 7.29–7.33 (1H, m, H Ar); 7.36–7.42 (2H, m, H Ar); 7.52–7.58 (2H, m, H Ar); 7.78–7.87 (4H, m, H Ar); 8.02 (1H, s, 16-CH); 8.25 (1H, d, *J* = 8.5, H Ar). ^{13}C NMR spectrum, δ, ppm: 45.5 (15-CH); 55.6 (OCH_3); 93.3 (7a-CH); 111.7; 114.7 (2CH); 115.4 (CH); 116.3; 117.4 (CH); 119.2 (CH); 121.9 (CH); 123.6 (CH); 124.4 (CH); 124.9 (CH); 127.5 (CH); 127.7 (CH); 129.1 (2CH); 129.4 (2CH); 129.6; 129.7; 129.9; 130.0; 130.3 (2CH); 132.1; 134.0; 147.7; 150.8; 158.8. Found, %: C 84.06; H 4.96. $C_{31}H_{22}O_3$. Calculated, %: C 84.14; H 5.01.

***trans*-15-(Thiophen-3-yl)-7a*H*,15*H*-benzo[*f*]benzo[5,6]-chromeno[2,3-*b*]chromene (3g)**. Yield 360 mg (86%), colorless crystals, mp 212–213°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 5.73 (1H, s, 15-CH); 6.63 (1H, s, 7a-CH); 7.04 (1H, br. s, H thiophene); 7.13 (1H, d, *J* = 8.9, H Ar); 7.17 (1H, d, *J* = 4.8, H thiophene); 7.26 (1H, d, *J* = 8.9, H Ar); 7.31–7.35 (1H, m, H Ar); 7.39–7.44 (2H, m, H Ar); 7.51

(H, dd, J = 4.8, J = 3.0, H thiophene); 7.55–7.59 (1H, m, H Ar); 7.62 (1H, d, J = 8.5, H Ar); 7.81–7.88 (4H, m, H Ar); 7.98 (1H, s, 16-CH); 8.23 (1H, d, J = 8.5, H Ar). ^{13}C NMR spectrum, δ, ppm: 42.1 (15-CH); 93.5 (7a-CH); 111.7; 115.9 (CH); 117.0; 117.4 (CH); 119.2 (CH); 121.9 (CH); 123.4 (2CH); 124.4 (CH); 124.9 (CH); 127.5 (2CH); 127.7 (CH); 128.2 (CH); 128.7; 129.1 (2CH); 129.6; 129.8; 130.0; 130.3 (CH); 130.4 (CH); 132.0; 143.0; 147.8; 150.3. Found, %: C 80.42; H 4.31; S 7.55. $C_{28}H_{18}O_2S$. Calculated, %: C 80.36; H 4.34; S 7.66.

7a*H*,10a*H*,18*H*,22*H*-Benzo[5,6]chromeno[2,3-*b*]benzo-[5',6']chromeno[3',2':5,6]pyrano[3,2-*g*]chromene (4). A mixture of 1*H*-benzo[*f*]chromene-2-carbaldehyde **1a** (210 mg, 1 mmol), resorcinol (55 mg, 0.5 mmol), $AcONH_4$ (155 mg, 2 mmol), and AcOH (5 ml) was heated under reflux for 4 h. After cooling the reaction mixture to room temperature, the formed precipitate was filtered off and recrystallized from AcOH. Yield 255 mg (52%), colorless crystals, mp 303–304°C. ^{1}H NMR spectrum, δ, ppm (J, Hz): 3.93 (2H, d, J = 18.1, CH_2); 4.06 (2H, d, J = 18.1, CH_2); 6.62 (2H, s, 7a,10a-CH); 6.75 (1H, s, H Ar); 6.80 (2H, s, 19,21-CH); 7.05 (2H, d, J = 8.7, H Ar); 7.08 (1H, s, H Ar); 7.36–7.40 (2H, m, H Ar); 7.51–7.55 (2H, m, H Ar); 7.72 (2H, d, J = 9.1, H Ar); 7.82–7.86 (4H, m, H Ar). ^{13}C NMR spectrum, δ, ppm: 30.6 ($2CH_2$); 95.5 (7a,10a-CH); 114.1; 115.3; 119.1; 122.9; 124.5; 124.8; 125.7; 127.4; 128.9; 132.3; 150.7; 150.8. Not all signals can be detected due to poor solubility in most organic solvents and low intensity of signals of carbon atoms in the ^{13}C NMR spectrum. Found, %: C 82.51; H 4.53. $C_{34}H_{22}O_4$. Calculated, %: C 82.58; H 4.48.

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