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I.P. Beletskaya on her jubilee

New Synthesis of 3-Amino-1*H*-benzo[*f*]chromene-2-carbonitriles

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Abstract—3-Amino-1*H*-benzo[*f*]chromene-2-carbonitriles were synthesized by non-catalytic reaction from Mannich bases of the naphthalene series and malononitrile. Reactive 1-benzylidene(or methyldiene)naphthalen-2(1*H*)-ones were presumed as intermediate products.

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Studies on the synthesis of fused aminochromenes are stimulated by diverse biological activity of these compounds. In particular, they exhibit antioxidant properties [1], while substituted 2-amino-4*H*-benzochromenes were proposed as medicines for the treatment of immune disorders and diabetes complications [2]. Some 3-amino-1*H*-benzo[*f*]chromene-2-carbonitriles were reported to possess anticancer [3] and antibacterial [4] activity. Furthermore, 3-amino-1*H*-benzo[*f*]chromene-2-carbonitriles may be regarded as heterocyclic β -enamino nitriles that are promising as building blocks for the synthesis of fused chromenes.

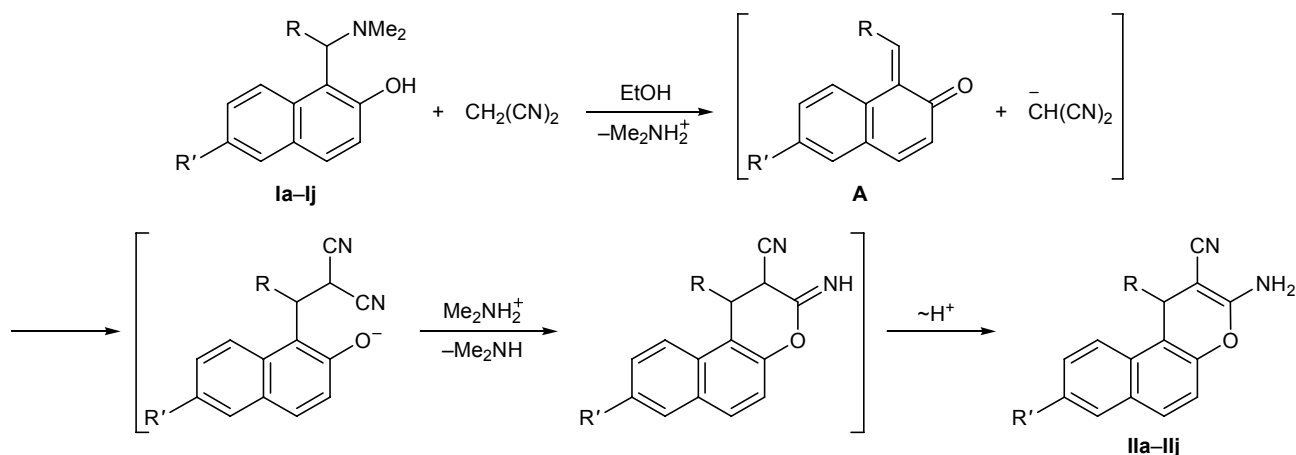
Several procedures for the synthesis of 3-amino-1*H*-benzo[*f*]chromene-2-carbonitriles have been reported. Among these, the most commonly used is three-component condensation of malononitrile with an aldehyde, primarily aromatic one, and naphthalen-2-ol. This reaction is generally carried out in ethanol, acetonitrile, or water in the presence of a base (K_2CO_3 [4], $NaHCO_3$ [5], triethylamine [6], DABCO [7]), acid ($TiCl_4$ [8], $H_{14}[NaP_5W_{30}O_{110}]$ [9]), or phase-transfer catalyst (cetyltrimethylammonium bromide [10] or chloride [11, 12], tetrabutylammonium bromide [13], sodium dodecanesulfonate [14]), as well as of ceric ammonium nitrate [3]. Another procedure includes preliminary preparation of alkylidenemalononitrile and its subsequent reaction with naphthalen-2-ol in the presence of piperidine [15–17] or sodium hydroxide [18] as base. 3-Amino-1*H*-benzo[*f*]chromene-2-carbonitrile having no substituent on C^1 was synthesized from 2-hydroxynaphthalene-1-carbaldehyde, malono-

nitrile, and the Hantzsch ester in the presence of $InCl_3$ [19]. Enantioselective synthesis of 3-amino-1*H*-benzo[*f*]chromene-2-carbonitriles from benzylidenemalononitriles was also reported [20].

We now propose a new procedure for the synthesis of 1-substituted 3-amino-1*H*-benzo[*f*]chromene-2-carbonitriles **IIa–IIj** from Mannich bases **Ia–Ij** of the naphthalene series and malononitrile. The reactions were carried out by heating equimolar amounts of the reactants in ethanol for 1 h, and compounds **IIa–IIj** were isolated in 72–90% yield. The nature of the R substituent does not affect the yield to a considerable extent. Apart from high yield and experimental simplicity, the proposed procedure is advantageous since there is no need of using additional catalyst. Moreover, the use of Mannich bases as starting compounds makes it possible to avoid side formation of 14*H*-dibenzo[*a,j*]-xanthenes, which frequently accompanies three-component condensations with naphthalen-2-ol [3, 21, 22].

The described reaction is a cascade process which is likely to include deamination of Mannich base with formation of intermediate *o*-quinone methide **A**, Michael addition of malononitrile, and intramolecular Pinner reaction [23] between the hydroxy and cyano groups. The subsequent proton migration yields final product **II**. Presumably, dimethylamine liberated in the first step favors deprotonation of malononitrile. We detected no 9,11-diamino-12*H*-benzo[5,6]chromeno-[2,3-*b*]pyridine-10-carbonitriles [24] that could be formed via addition of the second malononitrile molecule to benzochromenes **II**.

Scheme 1.



R' = H, R = H (**a**), Ph (**b**), 4-MeOC₆H₄ (**c**), 3-O₂NC₆H₄ (**d**), 2-FC₆H₄ (**e**), pyridin-4-yl (**f**), thiophen-2-yl (**g**), 1-benzyl-1*H*-imidazol-5-yl (**h**), 4-ClC₆H₄ (**i**); R' = adamantan-1-yl, R = H (**j**).

On the whole, quinone methides of the naphthalene series can be generated under considerably milder conditions than quinone methides of the benzene series, primarily due to extended conjugation chain in the former. Mannich base **1h** is converted into the corresponding *o*-quinone methides even at room temperature, as follows from the appearance of bright orange color upon dissolution of the colorless precursor in ethanol. Benzochromene **11h** is formed in 75% yield from compound **1h** and malononitrile in 48 h, whereas Mannich bases of the phenol series fail to react with malononitrile even on prolonged heating in boiling ethanol. This reaction becomes possible only when Mannich base ammonium salts are used as precursors of *o*-quinone methides in the presence of DBU or other bases [25].

Compounds **11a–11j** characteristically displayed in the IR spectra absorption bands in the region 2100–2200 cm^{−1} due to stretching vibrations of conjugated cyano group.

EXPERIMENTAL

The IR spectra were recorded in KBr on a Shimadzu FTIR-8400S spectrometer. The ¹H NMR spectra were measured on a JEOL JNM-ECX400 spectrometer (400 MHz) from solutions in CDCl₃ using tetramethylsilane as internal reference. The mass spectra (electron impact, 70 eV) were obtained on a Finnigan Trace DSQ instrument. The elemental compositions were determined on a EuroVector EA-3000 automatic CHNS analyzer. Previously described Mannich bases were synthesized from the corresponding

aldehyde, 33% aqueous dimethylamine, and naphthalen-2-ol (compounds **1a–1c**) [26, 27] or 6-(1-adamantyl)naphthalen-2-ol (**1j**) [28] in ethanol.

Compounds **1d–1i** were synthesized in a similar way.

1-[(1-Benzyl-1*H*-imidazol-5-yl)(dimethylamino)methyl]naphthalen-2-ol (1h). Naphthalen-2-ol, 3 g (0.021 mol), was dissolved in 15 ml of ethanol, 3.5 g (0.026 mol) of 33% aqueous dimethylamine and 3.9 g (0.021 mol) of 1-benzyl-1*H*-imidazole-5-carbaldehyde [29] were added, and the mixture was kept for 48 h at room temperature and for 2 h at −20°C. The precipitate was filtered off, washed with ice-cold methanol, and recrystallized from hot chloroform with addition of petroleum ether. Yield 5.4 g (72%), mp 227–229°C. IR spectrum, ν, cm^{−1}: 3100–2500 (OH), 1624, 1601, 1582, 1520, 1470, 1412, 1373, 1269, 1238, 1223, 1115, 953, 833, 710. ¹H NMR spectrum (CDCl₃), δ, ppm: 2.33 br.s [6H, (CH₃)₂N], 5.16 s (1H, CH), 5.29 s (2H, CH₂), 6.93–6.96 m (2H, H_{arom}), 7.02 d (1H, H_{arom}, *J* = 9.2 Hz), 7.07–7.11 m (1H, H_{arom}), 7.15–7.30 m (6H, H_{arom}), 7.41 s (1H, H_{arom}), 7.57 d (1H, H_{arom}, *J* = 8.7 Hz), 7.63 d (1H, H_{arom}, *J* = 7.8 Hz), 13.16 br.s (1H, OH). ¹³C NMR spectrum (CDCl₃), δ_C, ppm: 43.7 (CH₃), 49.2 (CH), 61.4 (CH₂), 113.6, 120.1, 120.7, 122.5, 126.5, 127.0, 128.3, 128.7, 128.9, 129.1, 130.0, 130.2, 131.8, 131.9, 135.7, 139.3, 155.0. Found, %: C 77.35; H 6.54; N 11.69. C₂₃H₂₃N₃O. Calculated, %: C 77.28; H 6.49; N 11.76.

1-[(Dimethylamino)(3-nitrophenyl)methyl]naphthalen-2-ol (1d). Yield 80%, mp 149–150°C (from MeOH). IR spectrum, ν, cm^{−1}: 3100–2500 (OH),

1620, 1601, 1528 (NO₂), 1470, 1354 (NO₂), 1269, 1238, 1188, 957, 814. ¹H NMR spectrum (CDCl₃), δ, ppm: 2.38 br.s [6H, (CH₃)₂N], 5.12 s (1H, CH), 7.19 d (1H, H_{arom}, *J* = 8.7 Hz), 7.36 d.d.d (1H, H_{arom}, *J* = 7.8, 6.9, 0.9 Hz), 7.41–7.45 m (2H, H_{arom}), 7.69–7.73 m (2H, H_{arom}), 7.83 d (1H, H_{arom}, *J* = 8.5 Hz), 7.98 d (1H, H_{arom}, *J* = 7.8 Hz), 8.07 d.d.d (1H, H_{arom}, *J* = 8.2, 2.3, 0.9 Hz), 8.46 d (1H, H_{arom}, *J* = 1.9 Hz), 13.38 br.s (1H, OH). ¹³C NMR spectrum (CDCl₃), δ_C, ppm: 44.7 (CH₃), 72.1 (CH), 115.3 (C_{arom}), 120.2 (CH_{arom}), 120.5 (CH_{arom}), 122.9 (CH_{arom}), 123.3 (CH_{arom}), 123.8 (CH_{arom}), 127.0 (CH_{arom}), 128.8 (C_{arom}), 129.2 (CH_{arom}), 130.2 (CH_{arom}), 130.3 (CH_{arom}), 131.9 (C_{arom}), 134.8 (CH_{arom}), 142.7 (C_{arom}), 148.4 (C_{arom}), 155.4 (C_{arom}). Found, %: C 70.85; H 5.59; N 8.74. C₁₉H₁₈N₂O₃. Calculated, %: C 70.79; H 5.63; N 8.69.

1-[(Dimethylamino)(2-fluorophenyl)methyl]naphthalen-2-ol (Ie). Yield 80%, mp 137–139°C (from MeOH). IR spectrum, ν, cm⁻¹: 3000–2500 (OH), 1624, 1601, 1520, 1480, 1450, 1269, 1242, 1092, 953, 825, 764. ¹H NMR spectrum (CDCl₃), δ, ppm: 2.26 s and 2.56 s [6H, (CH₃)₂N], 5.58 s (1H, CH), 7.01 t (1H, H_{arom}, *J* = 7.3 Hz), 7.09 t (1H, H_{arom}, *J* = 9.2 Hz), 7.16–7.23 m (2H, H_{arom}), 7.26 t (1H, H_{arom}, *J* = 7.3 Hz), 7.42 d.d.d (1H, H_{arom}, *J* = 8.7, 6.9, 1.4 Hz), 7.64 t.d (1H, H_{arom}, *J* = 7.8, 1.4 Hz), 7.72 d (1H, H_{arom}, *J* = 9.2 Hz), 7.73 d (1H, H_{arom}, *J* = 7.3 Hz), 7.85 d (1H, H_{arom}, *J* = 8.2 Hz). ¹³C NMR spectrum (CDCl₃), δ_C, ppm: 41.8 (CH₃), 45.5 (CH₃), 62.9 (CH), 115.3 d (CH_{arom}, *J* = 22.9 Hz), 115.6 (C_{arom}), 120.1 (CH_{arom}), 121.0 (CH_{arom}), 122.7 (CH_{arom}), 125.3 (CH_{arom}), 126.8 (CH_{arom}), 127.2 d (C_{arom}, *J* = 12.4 Hz), 128.7 (C_{arom}), 128.9 (CH_{arom}), 129.9 d (CH_{arom}, *J* = 8.6 Hz), 129.9 (CH_{arom}), 130.2 (CH_{arom}), 132.4 (C_{arom}), 156.3 (C_{arom}), 160.4 d (CF, *J* = 244.1 Hz). Found, %: C 77.30; H 6.20; N 4.66. C₁₉H₁₈FNO. Calculated, %: C 77.27; H 6.14; N 4.74.

1-[(Dimethylamino)(pyridin-4-yl)methyl]naphthalen-2-ol (If). Yield 80%, mp 164–166°C (from MeOH). IR spectrum, ν, cm⁻¹: 2800–2400 (OH), 1620, 1597, 1578, 1470, 1412, 1238, 949, 814, 752. ¹H NMR spectrum (CDCl₃), δ, ppm: 2.37 br.s [6H, (CH₃)₂N], 4.96 s (1H, CH), 7.16 d (1H, H_{arom}, *J* = 9.2 Hz), 7.25 d.d.d (1H, H_{arom}, *J* = 7.8, 6.9, 0.9 Hz), 7.41 d.d.d (1H, H_{arom}, *J* = 8.3, 6.9, 1.4 Hz), 7.52 d.d (2H, H_{arom}, *J* = 4.6, 1.4 Hz), 7.69 d (1H, H_{arom}, *J* = 8.7 Hz), 7.71 d (1H, H_{arom}, *J* = 8.3 Hz), 7.81 d (1H, H_{arom}, *J* = 8.7 Hz), 8.50 d.d (2H, H_{arom}, *J* = 4.6, 1.4 Hz). ¹³C NMR spectrum (CDCl₃), δ_C, ppm: 44.3 (CH₃), 71.9 (CH), 115.0, 120.1, 120.6, 122.8, 123.5, 126.8, 128.8, 129.2, 130.3,

131.9, 149.1, 150.5, 155.5. Found, %: C 77.74; H 6.47; N 9.98. C₁₈H₁₈N₂O. Calculated, %: C 77.67; H 6.52; N 10.06.

1-[(Dimethylamino)(thiophen-2-yl)methyl]naphthalen-2-ol (Ig). Yield 80%, mp 138–140°C (from MeOH). IR spectrum, ν, cm⁻¹: 3100–2300 (OH), 1620, 1597, 1470, 1323, 1265, 1238, 1188, 1157, 945, 818, 729. ¹H NMR spectrum (CDCl₃), δ, ppm: 2.39 br.s [6H, (CH₃)₂N], 5.30 s (1H, CH), 6.86 d.d (1H, H_{arom}, *J* = 5.0, 3.7 Hz), 7.14–7.21 m (2H, H_{arom}), 7.26 t (1H, H_{arom}, *J* = 7.3 Hz), 7.42 d.d (1H, H_{arom}, *J* = 8.4, 7.3 Hz), 7.68 d (1H, H_{arom}, *J* = 9.2 Hz), 7.33 d (1H, H_{arom}, *J* = 8.2 Hz), 7.92 d (1H, H_{arom}, *J* = 8.7 Hz). ¹³C NMR spectrum (CDCl₃), δ_C, ppm: 66.8 (CH), 116.6, 120.0, 120.9, 122.6, 125.8, 126.5, 126.7, 127.0, 128.7, 129.1, 129.9, 132.0, 143.3, 155.1. Found, %: C 71.94; H 6.11; N 5.04; S 11.40. C₁₇H₁₇NOS. Calculated, %: C 72.05; H 6.05; N 4.94; S 11.32.

1-[(4-Chlorophenyl)(dimethylamino)methyl]naphthalen-2-ol (Ii). Yield 80%, mp 138–140°C (from MeOH). IR spectrum, ν, cm⁻¹: 3100–2300 (OH), 1620, 1597, 1470, 1323, 1265, 1238, 1188, 1157, 945, 818, 729. ¹H NMR spectrum (CDCl₃), δ, ppm: 2.34 br.s [6H, (CH₃)₂N], 4.96 s (1H, CH), 7.16 d (1H, H_{arom}, *J* = 9.2 Hz), 7.22–7.26 m (3H, H_{arom}), 7.38 d.d.d (1H, H_{arom}, *J* = 8.7, 6.9, 1.4 Hz), 7.52 d.d (2H, H_{arom}, *J* = 6.9, 1.4 Hz), 7.68 d (1H, H_{arom}, *J* = 9.2 Hz), 7.71 d (1H, H_{arom}, *J* = 7.8 Hz), 7.79 d (1H, H_{arom}, *J* = 8.7 Hz). ¹³C NMR spectrum (CDCl₃), δ_C, ppm: 45.0 (CH₃), 72.2 (CH), 115.9, 120.1, 120.8, 122.6, 126.6, 128.8, 129.1, 129.9, 130.2, 132.0, 133.9, 138.9, 155.4. Found, %: C 73.14; H 5.88; N 4.55. C₁₉H₁₈ClNO. Calculated, %: C 73.19; H 5.82; N 4.49.

3-Amino-1-(1-benzyl-1*H*-imidazol-5-yl)-1*H*-benzo[*f*]chromene-2-carbonitrile (IIh). A mixture of 2 g (5.6 mmol) of Mannich base **Ih** and 0.37 g (5.6 mmol) of malononitrile in 10 ml of ethanol was heated for 1 h under reflux and was then kept at –20°C. The colorless precipitate was filtered off, washed with ice-cold ethanol, and recrystallized from EtOH–DMF. Yield 1.78 g (84%), mp 246–248°C (decomp.). IR spectrum, ν, cm⁻¹: 3500–2800 (NH₂), 2183 (CN), 1655, 1589, 1412, 1234, 1084, 1049, 825, 710. ¹H NMR spectrum (DMSO-*d*₆), δ, ppm: 5.27 s (1H, CH), 5.33 s (2H, CH₂), 6.27 s (1H, H_{arom}), 6.83 d (1H, H_{arom}, *J* = 8.5 Hz), 7.01–7.03 m (2H, H_{arom}), 7.07 br.s (2H, NH₂), 7.08–7.11 m (1H, H_{arom}), 7.17 d (1H, H_{arom}, *J* = 8.9 Hz), 7.28–7.34 m (4H, H_{arom}), 7.63 s (1H, H_{arom}), 7.79 d (2H, H_{arom}, *J* = 8.9 Hz). ¹³C NMR spectrum (DMSO-*d*₆), δ_C, ppm: 48.6 (CH),

55.9 (CH₂), 114.0, 117.1, 121.5, 123.3, 125.3, 127.4, 127.5, 127.9, 128.3, 128.9, 129.2, 130.2, 130.4, 131.2, 135.0, 137.3, 139.4, 147.5, 161.2, 161.3. Found, %: C 76.23; H 4.71; N 14.87. C₂₄H₁₈N₄O. Calculated, %: C 76.17; H 4.79; N 14.81.

Compounds **IIa–IIg**, **IIi**, and **IIj** were synthesized in a similar way.

3-Amino-1H-benzo[f]chromene-2-carbonitrile (IIa). Yield 76%, mp 209–211°C [25].

3-Amino-1-phenyl-1H-benzo[f]chromene-2-carbonitrile (IIb). Yield 90%, mp 279–281°C [20].

3-Amino-1-(4-methoxyphenyl)-1H-benzo[f]chromene-2-carbonitrile (IIc). Yield 72%, mp 195–196°C [20].

3-Amino-1-(3-nitrophenyl)-1H-benzo[f]chromene-2-carbonitrile (IId). Yield 79%, mp 234–235°C; published data [7]: mp 239–241°C.

3-Amino-1-(2-fluorophenyl)-1H-benzo[f]chromene-2-carbonitrile (IIe). Yield 89%, mp 278–279°C (decomp.); published data [7]: mp 294–295°C.

3-Amino-1-(pyridin-4-yl)-1H-benzo[f]chromene-2-carbonitrile (IIf). Yield 85%, mp 246–248°C (decomp.); published data [30]: mp 261–262°C.

3-Amino-1-(thiophen-2-yl)-1H-benzo[f]chromene-2-carbonitrile (IIg). Yield 79%, mp 257–258°C (decomp.) [31].

3-Amino-1-(4-chlorophenyl)-1H-benzo[f]chromene-2-carbonitrile (IIi). Yield 79%, mp 211–212°C [30].

8-(Adamantan-1-yl)-3-amino-1H-benzo[f]chromene-2-carbonitrile (IIj). Yield 74%, mp 268–269°C (decomp.). IR spectrum, ν , cm⁻¹: 3429 (NH₂), 3329 (NH₂), 2903 (CH, Ad), 2847 (CH, Ad), 2181 (CN), 1651, 1593, 1416, 1240, 1080, 802. ¹H NMR spectrum (DMSO-*d*₆), δ , ppm: 1.71 s (6H, CH₂, Ad), 1.90 s (6H, CH₂, Ad), 2.04 s (3H, CH, Ad), 3.71 s (2H, CH₂), 6.83 s (2H, NH₂), 7.10 d (1H, H_{arom}, *J* = 8.7 Hz), 7.63 d (1H, H_{arom}, *J* = 8.7 Hz), 7.71–7.73 m (2H, H_{arom}), 7.79 d (1H, H_{arom}, *J* = 9.2 Hz). ¹³C NMR spectrum (DMSO-*d*₆), δ _C, ppm: 22.2 (CH₂, Ad), 28.8 (CH, Ad), 36.3 (C, Ad), 36.7 (CH₂, Ad), 43.0 (CH₂), 49.8, 111.9, 116.9, 121.8, 123.3, 123.7, 125.8, 129.3, 129.5, 130.8, 146.3, 148.0, 160.8. Found, %: C 80.93; H 4.67; N 7.81. C₂₄H₂₄N₂O₂. Calculated, %: C 80.87; H 4.67; N 7.86.

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