Synthesis of new pyrido[3,4-*c*]carbazole derivatives

*V. M. Lyubchanskaya, L. M. Alekseeva, and V. G. Granik**

State Scientific Center of Antibiotics,
3a ul. Nagatinskaya, 117105 Moscow, Russian Federation.
E-mail: vggranik@mail.ru

Previously unknown 2-[6-hydroxy-9-(4-methoxyphenyl)-1,2,3,9-tetrahydro-4*H*-carbazol-4-ylidene]malononitrile was synthesized by the Nenitzescu reaction and was used for the construction of new tetracyclic compounds, *viz.*, pyrido[3,4-*c*]carbazoles.

Key words: Nenitzescu reaction, 6-hydroxy-4-dicyanomethylidenecarbazole, pyrido[3,4-*c*]carbazole.

The development of new approaches to the synthesis of new fused heterocycles is a topical problem of organic chemistry. Many compounds of the carbazole series fused to other heterocycles exhibit biological activity or are used as drugs.[1,2] 1-Oxotetrahydrocarbazole derivatives were used for annelation at positions 1, 9a, and 9,[1,2] as well as at positions 1 and 2.[3] The use of isomeric 4-oxo derivatives for the construction of heterocycles annulated at positions 3 and 4 of carbazole presents difficulties. Recently, it has been demonstrated[4] that 4-oxocarbazoles are less reactive than their 1-oxo analogs. The 4-oxo group provides the required level of CH-acidity of the adjacent methylene group to a much lesser degree, due to which, for example, DMF acetal cannot be fused at this methylene unit.[4] Hence, tetrahydrocarbazol-4-ones are not very attractive as components for the construction of new tetracyclic systems by annulation at the 3,4 bond.

We succeeded in synthesizing tetrahydrocarbazole containing the dicyanomethylene group at position 4. We expected that the presence of this substituent containing two electron-withdrawing cyano groups would provide better activation of the adjacent 3-CH_2 group toward the nucleophilic attack than the carbonyl group.

To synthesize this carbazole by the Nenitzescu reaction, we used for the first time new enamine, *viz.*, [3-(4-methoxyanilino)-2-cyclohexenylidene]malononitrile (**1**), which was prepared from (3-oxocyclohexyl-1-idene)malononitrile[5] and *p*-anisidine. Condensation of *p*-benzoquinone with enamine **1** in acetone in the presence of TsOH leads to indole cyclization giving rise to 2-[6-hydroxy-9-(4-methoxyphenyl)-1,2,3,9-tetrahydro-4*H*-carbazol-4-ylidene]malononitrile (**2**) in a yield (40%) satisfactory for the Nenitzescu reaction (Scheme 1).

To prevent side reactions associated, for example, with bromination of the benzene ring activated by the hydroxy group or alkylation of the phenol group in compound **1** typical of amide acetals,[6] we prepared *O*-acetyl derivative **3**. It appeared that bromination of acetoxycarbazole **3** occurs unusually to give 7-bromo derivative **4** (rather than the expected 3-bromo derivative) regardless of the nature of the brominating agent (bromine or *N*-bromosuccinimide) and the solvent (AcOH, CCl_4, or CH_2Cl_2). The following two unusual facts should be noted: the methylene group at position 3 of carbazole is inert toward bromination, and the reaction proceeds at the benzene ring although it is known that, in the presence of the *O*-acetoxy group in indole or benzofuran, the side chain is generally subjected to bromination.[7,8] The reactions of carbazoles **3** and **4** with DMF diethyl acetal produce the corresponding 3-dimethylaminomethylidene derivatives **5** and **6** that serve as the key compounds for further heterocyclizations.

It should be noted that condensation at the active methylene unit of tricyclic compound **2** requires the use of triethylamine as the catalyst, which is, as a rule, not necessary in the reactions of amide acetals with active methylene compounds. In our opinion, this is due to the above-mentioned relatively low CH-acidity of the methylene unit at position 3 of the tetrahydrocarbazole molecule. Even in the presence of the dicyanomethylidene fragment, the reaction of the fused indole ring adversely affects the possibility of generating the 3-CH anion due to resonance, which leads to a substantial weakening of the electronegative effect of the cyano groups (Scheme 2).

It should be noted that the presence of even a rather weak electron-withdrawing group, such as the bromine atom, at position 7 of the carbazole molecule makes it possible to perform the reaction of compound **6** with DMF acetal without the use of triethylamine as the catalyst.

It is known[6,9] that high reactivity of amide acetals is based on the fact that in solution these compounds exist in equilibrium with alkoxy anions and immonium cations, the equilibrium being, however, strongly shifted to the left[10] (Scheme 3).

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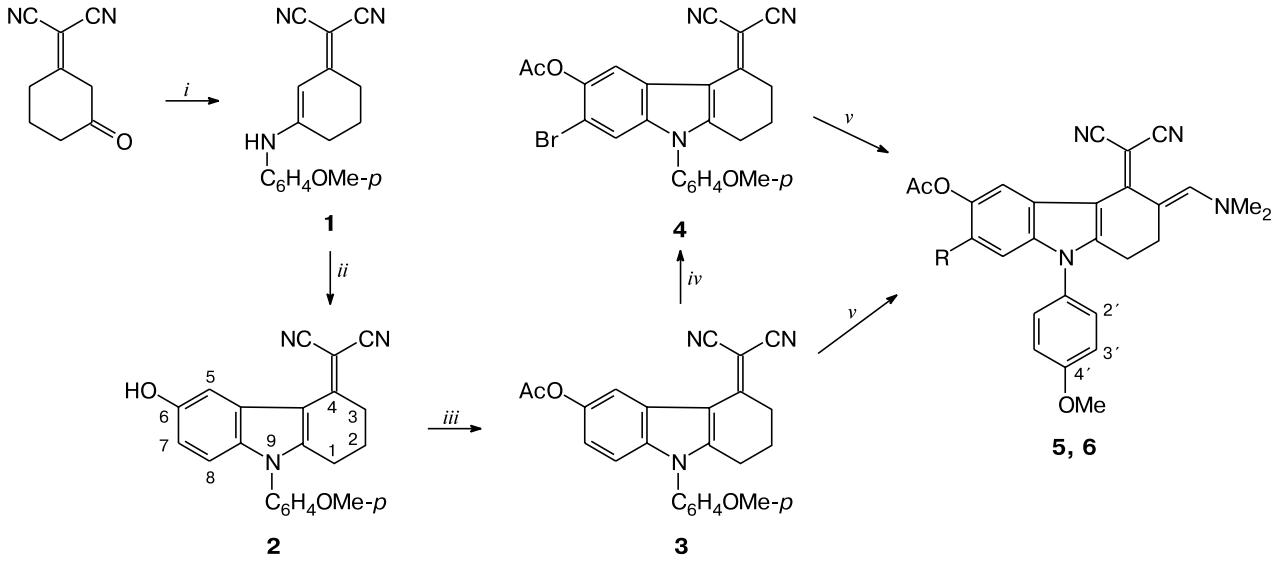
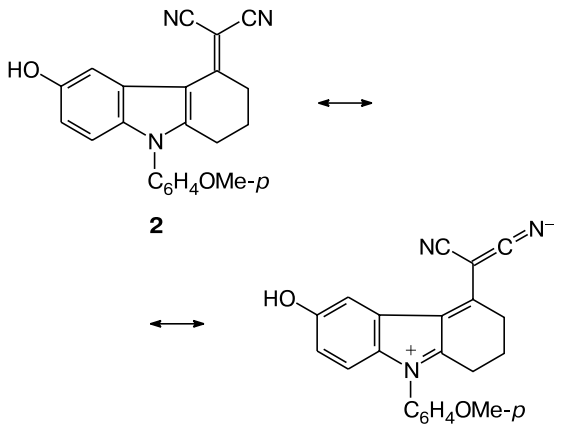
Scheme 1



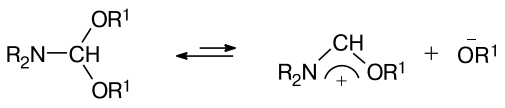
R = H (**5**), Br (**6**)

Reagents and conditions: *i*. *p*-anisidine, TsOH, benzene, refluxing, 7 h; *ii*. *p*-benzoquinone, TsOH, acetone, 20 °C, 8 h; *iii*. Ac_2O, H_2SO_4; *iv*. Br_2, CH_2Cl_2, refluxing, 5.5 h; *v*. DMF diethyl acetal, toluene, triethylamine, refluxing, 15 min.

Scheme 2



Scheme 3



As a result, in the case of low CH-acidity of the active methylene group, it is necessary to add an additional basic catalyst facilitating proton abstraction from the CH_2 group.

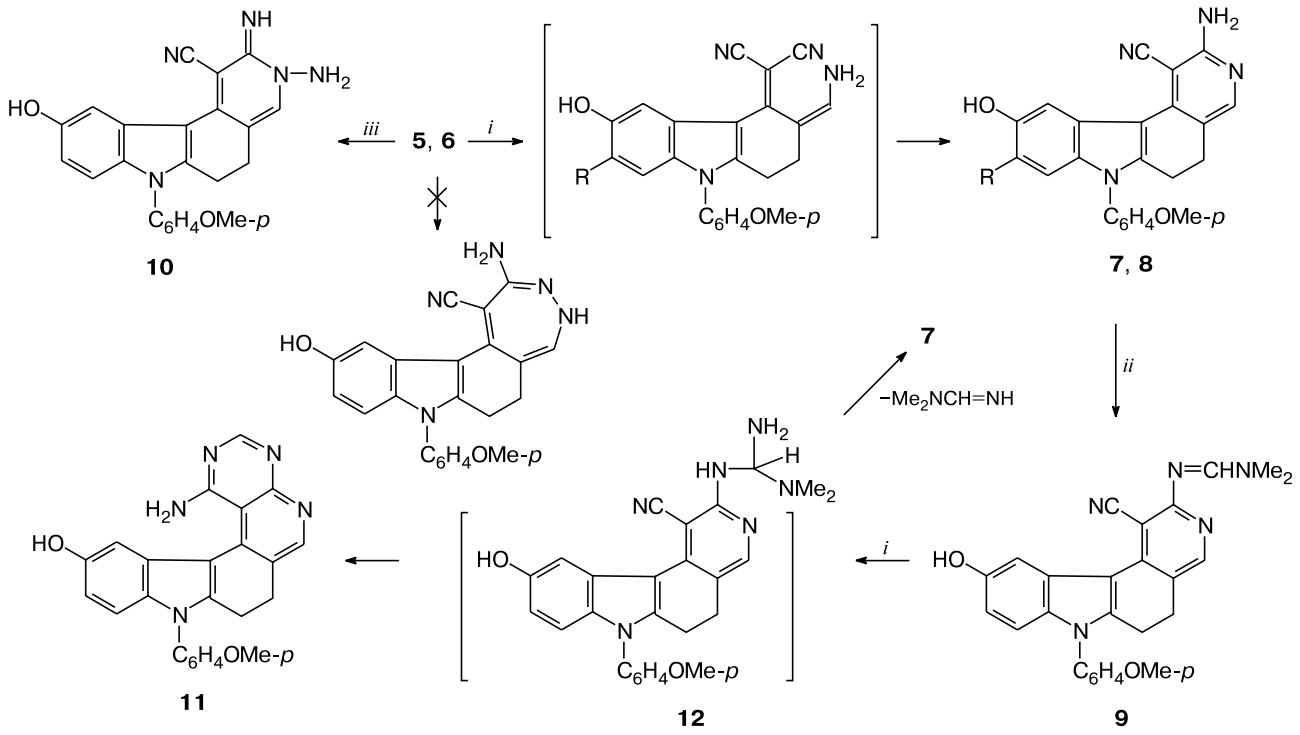
Compounds **5** and **6** contain functional substituents (the nitrile group and the enamine fragment) in the adjacent positions, which allows annulation of another ring. This type of cyclizations was described in detail in the review.[6] In some cases, such cyclizations led to the construction of the pyridine and pyrimidine rings.[6] In the present study, we used this approach for the synthesis of new tetracyclic compounds **7—10** and pentacyclic compound **11**. Treatment of enamines **5** and **6** with ammonium acetate gives rise to transamination accompanied by pyridine cyclization and *O*-deacetylation to form pyrido[3,4-*c*]carbazole derivatives **7** and **8**. The reaction of carbazole **7** with DMF diethyl acetal proceeds smoothly to give amidine **9** (Scheme 4).

We expected that treatment of enamine **5** with hydrazine hydrate would afford the 1,2-diazepinocarbazole derivative. However, this attempt also led to pyridine cyclization giving rise to *N*-aminopyridine derivative **10**. Due to the presence of two adjacent functional groups in compound **9**, we subjected the latter compound to yet another cyclization. The reaction of amidine **9** with ammonium acetate produced pentacyclic pyrimidopyridocarbazole derivative **11** in 24% yield. The low yield is due to the fact that pyridocarbazole **7** was isolated as the major reaction product.

The formation of compound **7** along with pentacyclic compound **11** is attributed to the fact that intermediate **12** in this transformation is consumed for both cyclization to form compound **11** and fragmentation yielding *N*,*N*-dimethylformamidine and pyridocarbazole **7**.

The structures of the resulting compounds were established by mass spectrometry and NMR spectroscopy. Based on the ^1H NMR spectrum of compound **10**, it was impossible to unambiguously decide whether this com-

Scheme 4



R = H (**7**), Br (**8**)

Reagents and conditions: *i*. ammonium acetate, 140 °C, 30 min, silica gel chromatography; *ii*. DMF diethyl acetal, Pr^{i}OH, refluxing, 15 min; *iii*. hydrazine hydrate, MeOH, 20 °C, 12 h.

pound has the diazepine or pyridine structure, because the spectroscopic data do not contradict both these structures. Structure **10** is evidenced by electrospray and electron impact mass spectra. Both mass spectra of compound **10** show intense peaks at 381 $[M - 16]^+$, which indicates that the NH_2 group is easily eliminated, and this process is possible only for *N*-aminopyridine **10**.

Experimental

The electrospray mass spectra were recorded on a Waters ZQ-2000 mass spectrometer using a system for injection of the sample without a chromatographic column and on a Finnigan SSQ-710 mass spectrometer using a direct inlet system. The ^{1}H NMR spectra were measured on a Bruker AC-300 spectrometer in DMSO-d_6 with the use of the standard Bruker software. The course of the reactions was monitored and the purity of the compounds was checked on Merck 60 F_{254} plates. The yields, elemental analysis data, and physicochemical characteristics are given in Table 1.

[3-(4-Methoxyphenylamino)-2-cyclohexenylidene]malononitrile (1). A mixture of (3-oxocyclohexyl-1-idene)malononitrile[5] (7.73 g, 48 mmol), *p*-anisidine (5.94 g, 48 mmol), and TsOH (0.001 g) in benzene (400 mL) was refluxed with a Dean—Stark trap for 7 h and then kept at 20 °C for 12 h. The precipitate was filtered off, washed with benzene, dried, and recrystallized from benzene. Compound **1** was obtained in a yield of 11.2 g. ^{1}H NMR (DMSO-d_6), δ: 1.84 (br.s, 2 H, H(5); 2.59 (m, 4 H, 2 H(4), 2 H(6)); 3.77 (s, 3 H, OMe); 5.80 (s, 1 H, H(2)); 7.01 (m, 2 H, H(3′), H(5′)); 7.18 (m, 2 H, H(2′), H(6′)); 10.05 (br.s, 1 H, NHC(3)).

[6-Hydroxy-9-(4-methoxyphenyl)-1,2,3,9-tetrahydro-4*H*-carbazol-4-ylidene]malononitrile (2). Compound **1** (10.6 g, 40 mmol) and TsOH (6.88 g, 40 mmol) were added with stirring to a solution of *p*-benzoquinone (4.32 g, 40 mmol) in acetone (120 mL) at 20 °C. The reaction mixture was stirred for 8 h, and the precipitate was filtered off, refluxed in acetone, and again filtered off. The combined acetone filtrates were concentrated, the residue was triturated with hot ethanol, and cooled, and the precipitate was filtered off, washed with ethanol, and dried. Compound **2** was obtained in a yield of 5.68 g. ^{1}H NMR (DMSO-d_6), δ: 2.01 (q, 2 H, 2 H(2), J_o = 6.0 Hz); 2.74 and 2.93 (both t, 2 H each, 2 H(1), 2 H(3), J_o = 6.0 Hz); 3.85 (s, 3 H, OMe); 6.75 (dd, 1 H, H(7), J_o = 8.8 Hz, J_m = 2.2 Hz); 6.89 (d, 1 H, H(8), J_o = 8.8 Hz); 7.17 (m, 2 H, H(3′), H(5′)); 7.37 (d, 1 H, H(5), J_m = 2.2 Hz); 7.45 (m, 2 H, H(2′), H(6′)); 9.39 (br.s, 1 H, OH).

[6-Acetoxy-9-(4-methoxyphenyl)-1,2,3,9-tetrahydro-4*H*-carbazol-4-ylidene]malononitrile (3). One drop of sulfuric acid was added to a suspension of compound **2** (7.11 g, 20 mmol) in

Table 1. Yields, melting points, elemental analysis data, and mass spectrometric data for compounds **1—11**

Compound	Yield (%)	M.p./°C (solvent)	Found / Calculated (%)			Molecular formula	MS (electrospray ionization)
			C	H	N		
1	88	135—138 (benzene)	72.87 72.43	5.70 5.70	15.60 15.84	$C_{16}H_{15}N_3O$	266 $[M + H]^+$, 553 $[2 M + Na]^+$, 818 $[3 M + Na]^+$
2	40	243—245 (EtOH)	74.34 74.35	5.30 4.82	11.84 11.82	$C_{22}H_{17}N_3O_2$	356 $[M + H]^+$, 378 $[M + Na]^+$, 733 $[2 M + Na]^+$
3	84	183—185 (EtOH)	72.49 72.53	4.93 4.82	10.50 10.57	$C_{24}H_{19}N_3O_3$	397 $[M + H]^+$, 420 $[M + Na]^+$, 817 $[2 M + Na]^+$
4	63	180—183 (toluene)	60.36 60.51	4.04 3.81	8.73 8.82	$C_{24}H_{18}BrN_3O_3$	500 $[M + Na]^+$, (Br^{81}); 541 $[M + Na + MeCN]^+$, (Br^{81})
5	70.8	252—255 (Pr^iOH)	71.68 71.66	5.38 5.35	12.37 12.38	$C_{27}H_{24}N_4O_3$	452 $[M + H]^+$, 474 $[M + Na]^+$, 490 $[M + K]^+$, 927 $[2 M + Na]^+$
6	48	230—233 (Pr^iOH)			11.00 10.54	$C_{27}H_{23}BrN_4O_3$	530 $[M + H]^+$, (Br^{79}); 533 $[M + H]^+$, (Br^{81})
7	50	287—290 (dichloroethane)	72.33 72.23	4.72 4.74	14.44 14.65	$C_{23}H_{18}N_4O_2$	383 $[M + H]^+$, 405 $[M + Na]^+$, 787 $[2 M + Na]^+$
8	27	269—271 (MeCN)	59.35 59.88	3.74 3.71	11.65 12.14	$C_{23}H_{17}BrN_4O_2$	499 $[M + K]^+$, (Br^{79}); 501 $[M + K]^+$, (Br^{81})
9	68.4	258—262 (Pr^iOH)	70.80 71.38	4.74 5.30	15.52 16.00	$C_{26}H_{23}N_5O_2$	438 $[M + H]^+$, 875 $[2 M + H]^+$, 897 $[2 M + Na]^+$
10	94.7	275 decomp. (DMF)	69.78 69.51	4.85 4.82	17.23 17.62	$C_{23}H_{18}N_5O_2$	398 $[M + H]^+$, 795 $[2 M + Na]^+$
11	24.4	293—295 (DMF)	69.94 70.40	4.85 4.68	17.23 17.11	$C_{23}H_{18}N_5O_2$	398 $[M + H]^+$, 795 $[2 M + Na]^+$

acetic anhydride (65 mL) and the mixture was heated until a solution was obtained. Then the mixture was cooled and the precipitate that formed was filtered off, washed with water, and dried. Compound **3** was obtained in a yield of 6.68 g. ^{1}H NMR (DMSO-d_6), δ: 2.05 (q, 2 H, 2 H(2), J_o = 6.0 Hz); 2.29 (s, 3 H, OAc); 2.79 and 2.96 (both t, 2 H each, 2 H(1), 2 H(3), J_o = 6.0 Hz); 3.87 (s, 3 H, OMe); 7.05 (dd, 1 H, H(7), J_o = 8.8 Hz, J_m = 1.9 Hz); 7.11 (d, 1 H, H(8′), J_o = 8.8 Hz); 7.19 (m, 2 H, H(3′), H(5′)); 7.50 (m, 2 H, H(2′), H(6′)); 7.72 (d, 1 H, H(5), J_m = 1.9 Hz).

[6-Acetoxy-7-bromo-9-(4-methoxyphenyl)-1,2,3,9-tetrahydro-4*H*-carbazol-4-ylidene]malononitrile (4). A solution of bromine (1.12 g, 7 mmol) in dichloromethane (50 mL) was added dropwise with stirring to a boiling solution of compound **3** (2.85 g, 7 mmol) in dichloromethane (35 mL) for 1.5 h. Then the mixture was refluxed with stirring for 3 h. The solvent was removed, the residue was triturated with ethanol, and the precipitate was filtered off and recrystallized from toluene. Compound **4** was obtained in a yield of 2.1 g. ^{1}H NMR (DMSO-d_6), δ: 2.07 (q, 2 H, 2 H(2), J_o = 6.0 Hz); 2.35 (s, 3 H, OAc); 2.78 and 2.96 (both t, 2 H each, 2 H(1), 2 H(3′), J_o = 6.0 Hz); 3.88 (s, 3 H, OMe); 7.20 (m, 2 H, H(3′), H(5′)); 7.31 (s, 1 H, H(5)); 7.51 (m, 2 H, H(2′), H(6′)); 7.89 (s, 1 H, H(8)).

[6-Acetoxy-3-(dimethylamino)methylidene-9-(4-methoxyphenyl)-1,2,3,9-tetrahydro-4*H*-carbazol-4-ylidene]malononitrile (5). Dimethylformamide diethyl acetal (5.4 mL, 3.7 mmol) and triethylamine (1 drop) were added to a suspension of compound **3** (1.47 g, 3.7 mmol) in toluene (35 mL). The reaction mixture was refluxed for 15 min and then cooled. The precipitate was filtered off, washed with toluene, and dried. Compound **5** was obtained in a yield of 1.06 g. ^{1}H NMR (DMSO-d_6), δ: 2.30 (s, 3 H, OAc); 2.64 (s, 4 H, 2 H(1), 2 H(2)); 3.27 (s, 6 H, NMe_2); 3.86 (s, 3 H, OMe); 7.00 (dd, 1 H, H(7), J_o = 8.8 Hz, J_m = 2.1 Hz); 7.15 (m, 2 H, H(3′), H(5′)); 7.18 (d, 1 H, H(8), J_o = 8.8 Hz); 7.47 (m, 2 H, H(2′), H(6′)); 7.57 (d, 1 H, J_m = 2.1 Hz, H(5)); 8.02 (s, 1 H, CHN).

[6-Acetoxy-7-bromo-3-(dimethylamino)methylidene-9-(4-methoxyphenyl)-1,2,3,9-tetrahydro-4*H*-carbazol-4-ylidene]malononitrile (6). The synthesis was carried out analogously to that of compound **5** but without triethylamine. Compound **6** was obtained in a yield of 1.0 g from compound **4** (1.9 g, 4 mmol). ^{1}H NMR (DMSO-d_6), δ: 2.35 (s, 3 H, OAc); 2.63 (s, 4 H, 2 H(1), 2 H(2)); 3.28 (s, 6 H, NMe_2); 3.86 (s, 3 H, OMe); 7.18 and 7.50 (both m, 2 H each, C_6H_4OMe); 7.3 and 7.70 (both s, 1 H each, H(5), H(8)); 8.06 (s, 1 H, H(1)).

2-Amino-10-hydroxy-7-(4-methoxyphenyl)-6,7-dihydro-5*H*-pyrido[3,4-*c*]carbazole-1-carbonitrile (7). A mixture of compound **5** (2.05 g, 4.5 mmol) and ammonium acetate (35 g, 45 mmol) was heated at 135—140 °C for 30—35 min. After cooling, the reaction mixture was diluted with water, and the precipitate was filtered off, washed with water, dried, suspended in hot dichloroethane, and chromatographed on a silica gel column eluted with ethyl acetate. Compound **7** was obtained in a yield of 0.86 g. ^{1}H NMR (DMSO-d_6), δ: 2.65 (s, 4 H, 2 H(5), 2 H(6)); 3.85 (s, 3 H, OMe); 6.50 (br.s, 2 H, NH_2); 6.70 (dd, 1 H, H (9), J_o = 8.8 Hz, J_m = 2.4 Hz); 6.98 (d, 1 H, H(8), J_o = 8.8 Hz); 7.15 (m, 2 H, H(3), H(5)); 7.42 (m, 3 H, H(2), H (6), H (11)); 7.99 (s, 1 H, H(4)); 9.09 (br.s, 1 H, OH).

2-Amino-11-bromo-10-hydroxy-7-(4-methoxyphenyl)-6,7-dihydro-5*H*-pyrido[3,4-*c*]carbazole-1-carbonitrile (8). The syn-

thesis was carried out analogously to that of compound **7**. Compound **8** was obtained in a yield of 0.1 g from compound **6** (0.43 g, 0.8 mmol). ^{1}H NMR (DMSO-d_6), δ: 2.66 (s, 4 H, 2 H(5), 2 H(6)); 3.86 (s, 3 H, OMe); 6.54 (br.s, 2 H, NH_2); 7.17 (m, 2 H, H(3), H(5)); 7.21 (s, 1 H, H(11)); 7.46 (m, 2 H, H(2), H(6)); 7.63 (s, 1 H, H(8)); 8.01 (s, 1 H, H(4)); 9.94 (br.s, 1 H, OH).

1-Cyano-2-(dimethylamino)methylideneamino-10-hydroxy-7-(4-methoxyphenyl)-6,7-dihydro-5*H*-pyrido[3,4-*c*]carbazole (9). A suspension of compound **7** (0.17 g, 0.44 mmol) and dimethylformamide diethyl acetal (0.65 mL, 4.4 mmol) in isopropyl alcohol (4 mL) was refluxed for 15 min. The reaction mixture was cooled, abd the precipitate was filtered, washed with cold isopropyl alcohol, and dried. Compound **9** was obtained in a yield of 0.13 g. ^{1}H NMR (DMSO-d_6), δ: 2.73 (s, 4 H, 2 H(5), 2 H(6)); 3.09 and 3.15 (both s, 3 H each, NMe_2); 3.85 (s, 3 H, OMe); 6.68 (dd, 1 H, H (9), J_o = 8.4 Hz, J_m = 2.1 Hz); 7.00 (d, 1 H, H(8), J_o = 8.4 Hz); 7.15 (m, 2 H, H(3′), H(5′)); 7.44 (m, 3 H, H(11), H(2′), H(6′)); 8.15 (s, 1 H, H(4)); 8.63 (s, 1 H, H(1)); 9.07 (br.s, 1 H, N(4)H).

3-Amino-10-hydroxy-2-imino-7-(4-methoxyphenyl)-3,5,6,7-tetrahydro-2*H*-pyrido[3,4-*c*]carbazole-1-carbonitrile (10). A suspension of compound **5** (0.45 g, 1 mmol) and hydrazine hydrate (0.15 mL, 3 mmol) in methanol (25 mL) was stirred at 20 °C for 12 h. The precipitate was filtered off, washed with methanol, and dried. Compound **10** was obtained in a yield of 0.38 g. ^{1}H NMR (DMSO-d_6), δ: 2.53 and 2.66 (both t, 2 H each, 2 H(6), 2 H(7), J_o = 6.9 Hz); 3.86 (s, 3 H, OMe); 5.83 (br.s, 2 H, NH_2); 6.71 (dd, 1 H, H(10), J_o = 8.8 Hz, J_m = 2.1 Hz); 6.96 (d, 1 H, H(9), J_o = 8.8 Hz); 7.15 (m, 2 H, H(3), H(5)); 7.40 (m, 3 H, H(12), H(2), H (6)); 7.58 (s, 1 H, H(5)); 9.18 (br.s, 1 H, OH).

1-Amino-12-hydroxy-9-(4-methoxyphenyl)-8,9-dihydro-7*H*-pyrimido[5′,4′:4,5]pyrido[2,3-*c*]carbazole (11). A mixture of compound **9** (0.87 g, 2 mmol) and ammonium acetate (15.4 g, 200 mmol) was heated at 135—140 °C for 1 h, cooled, and diluted with water. The precipitate was filtered off, washed with water, dried, suspended in hot dichloroethane, and chromatographed on a silica gel column. Elution with ethyl acetate afforded compound **7** in a yield of 0.44 g (57%), and then elution with methanol gave compound **11** in a yield of 0.2 g. ^{1}H NMR (DMSO-d_6), δ: 2.70 and 2.97 (both m, 1 H each, 2 H(8), 2 H(7); 3.86 (s, 3 H, OMe); 6.37 and 7.99 (both br.s, 1 H each, NH_2); 6.68 (dd, 1 H, H(11), J_o = 8.8 Hz, J_m = 2.2 Hz); 6.79 (d, 1 H, H(13), J_m = 2.2 Hz); 7.05 (d, 1 H, H(10), J_o = 8.8 Hz); 8.42 (s, 1 H, H(5)); 8.69 (s, 1 H, H(3)); 9.12 (br.s, 1 H, OH).

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