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Synthesis of 1-(1-Aryl-1*H*-1,2,3-triazol-4-yl)-β-carboline Derivatives

N. T. Pohodylo, V. S. Matiichuk, and M. D. Obushak

Ivan Franko Lviv National University, Lviv, 79005 Ukraine
e-mail: obushak@in.lviv.ua

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Abstract—Reaction of 5-methyl-1-aryl-1*H*-1,2,3-triazole-4-carbocylic acid chlorides with tryptamine derivatives afforded substituted 1-aryl-*N*-[2-(1*H*-indol-3-yl)ethyl]-5-methyl-1*H*-1,2,3-triazole-4-carboxamides. At heating these compounds in toluene in the presence of $POCl_3$ and P_2O_5 Bischler-Napieralski cyclization occurs giving 1-(1-aryl-5-methyl-1H-1,2,3-triazol-4-yl)-4,9-dihydro-3H-β-carbolines that can be transformed into β-carboline and tetrahydro-β-carboline derivatives.

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Heterocyclic system of β-carboline (9*H*-pyrido[3,4-*b*]-indole) is an important structural fragment that is often present in alkaloids and their synthetic analogs possessing biological activity [1, 2]. In this connection special attention is attracted by the methods of isolation and preparation of either natural or synthetic derivatives of β-carboline [3–7]. The accesibility of this heterocyclic system is due, first of all, to the synthetic opportunities provided by Pictet-Spengler and Bischler–Napieralski reactions [8–10]. The last reaction posesses higher diversity and utilizes more accessible initial reagents. The dihydro-β-carbolines obtained are easily oxidized to β-carbolines or reduced to tetrahydro-β-carbolines.

Bischler–Napieralski reaction is widely used in the synthetic practice and involves the cyclodehydration of (het)arylethylamides [9]. Its assumed mechanism involves the formation of intermediate imido chlorides or nitrilium ions, which is confirmed by some Ritter retroreactions [11], and the subsequent intramolecular electrophilic aromatic substitution. The reaction is applicable to a wide range of compounds with substituents in aromatic ring, in α- and β-positions of ethylamine fragment, and also to various substituted amides [12].

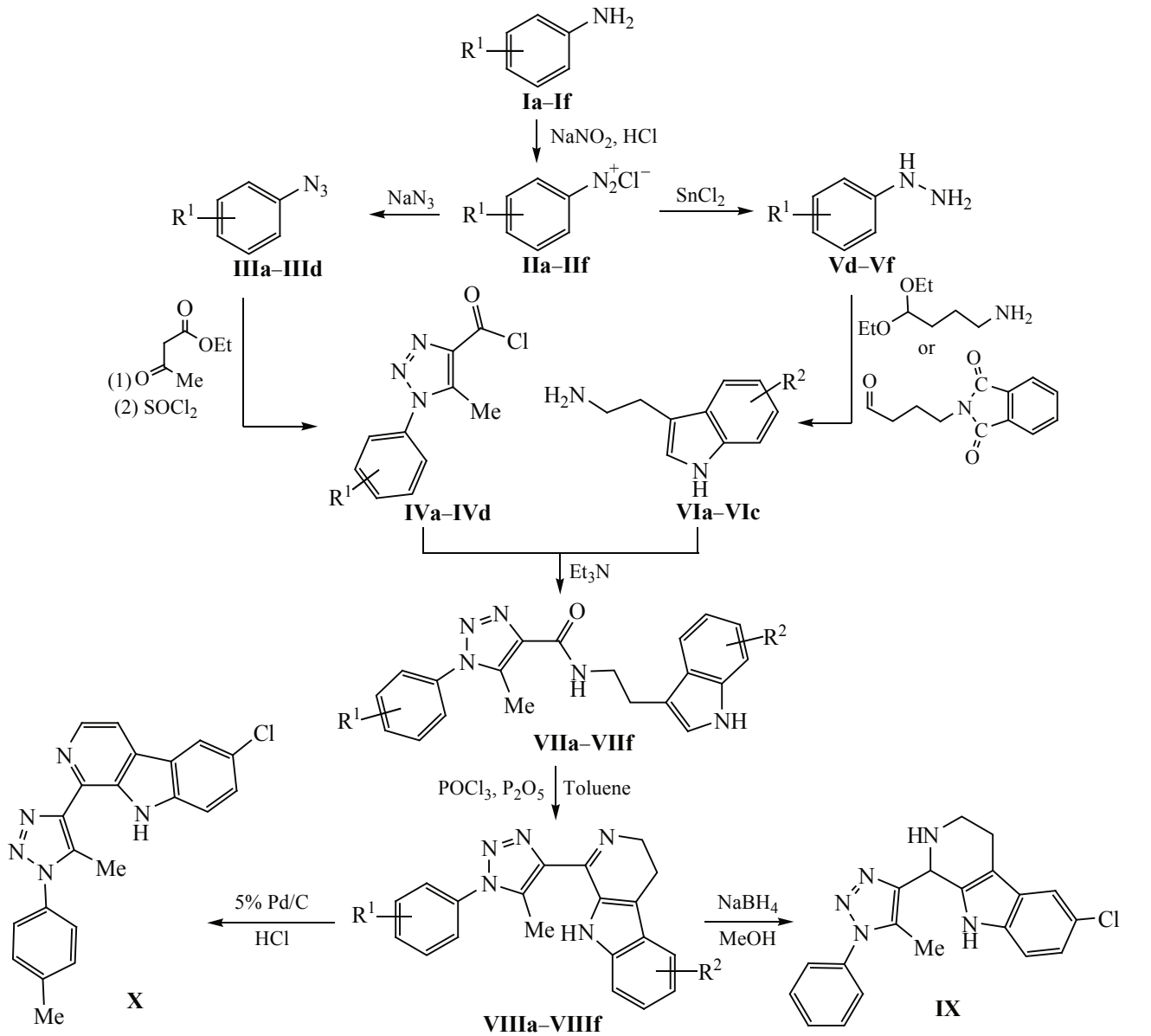
In classical version the reaction is carried out in boiling solvent (e.g., in toluene) with the use of $POCl_3$ or P_2O_5. In the syntheses of biologically active compounds by Bischler–Napieralski reaction $POCl_3$ [13–16], P_2O_5 [17], and $POCl_3$ in ionic liquids [18] were used as reagents. The severe conditions may be the reason of complications occurring in the course of these reactions, especially in presence of reactive functional groups. In these cases softer conditions were developed utilizing in Bischle–Napieralski cyclisation triphosgene [19], also the systems of oxalyl chloride–$(COCl)_2$–$FeCl_3$ in dichloromethane [20], triphenylphosphine in boiling CCl_4 [21], Tf_2O–DMAP [22] or bromotriphenoxyphosphonium bromide $(PhO)_3PBr_2$ [23].

While the cyclization is an electophilic aromatic substitution, the substrates with electon-donor substituents give as a rule better results. The compounds that do not contain electron-donor groups often do not undergo cyclization or give final product in small yields.

In the present study we investigated the possibility to obtain by Bischler–Napieralski reaction β-carboline derivatives with 1,2,3-triazole substituents, using as initial substances substituted 1,2,3-triazole-4-carbocylic acids. Considering the results of our previous investigations [24] we could have expected probable complications connected with the electron-donor effect of the triazole ring on the reactivity of the carboxy and amide groups, with the formation of reactive intermediates, and proceeding of competitive processes. 1,2,3-Triazole-4-carboxylic acids have never been studied before in such reactions.

Initial reagents were obtained from commercially available aromatic amines and diazonium salts on their basis. Aryl azides **IIIa–IIId** were obtained by diazotiza-

tion of arylamines **Ia–Id** followed by treating the diazonium salts **IIa–IId** with potassium azide. The cyclization of ethyl acetoacetate with aryl azides **IIIa–IIId** leads to the formation of 1-aryl-5-methyl-1*H*-1,2,3-triazole-4-carboxylic acids that are transformed into acyl chlorides **IVa–IVd** [24]. All tryptamine derivatives **VIa–VIc** used in our investigation were synthesized by Fischer method. The arylhydrazines **Vd–Vf** obtained by reduction of the diazonium salts **IId–IIf** with $SnCl_2$ were brought into the reaction with the 4-aminobutanal acetal or with 4-phtalamidobutanal with subsequent removal of the protection [25–28]. Compounds **VIa–VIc** smoothly underwent acylation with acid chlorides **IVa–IVd**, and as a result amides **VIIa–VIIf** were obtained that then were tested in Bischler–Napieralski cyclization. As the experiment demonstrated, the cyclization took place when the reaction was performed in acetontrile in the presence of $POCl_3$, but compounds **VIIIa–VIIIf** were obtained in low



I–V, R^1 = H (**a**), 4-Me (**b**), 4-MeO (**c**), 4-F (**d**), 4-Cl (**e**), 3-MeO (**f**); **VI**, R^2 = 5-F (**a**), 5-Cl (**b**), 6-MeO (**c**); **VII**, R^1 = H, R^2 = 6-MeO (**a**), 5-F (**b**), 5-Cl (**c**); R^1 = 4-F, R^2 = 6-MeO (**d**); R^1 = 4-MeO, R^2 = 5-F (**e**); R^1 = 4-Me, R^2 = 5-Cl (**f**); **VIII**, R^1 = H, R^2 = 7-MeO (**a**), 6-F (**b**), 6-Cl (**c**); R^1 = 4-F, R^2 = 7-MeO (**d**); R^1 =4-MeO, R^2 = 6-F (**e**); R^1 = 4-Me, R^2 = 6-Cl (**f**).

or medium yields. Rising the temperature of the reaction to 110°C and bringing P_2O_5 into the reaction mixture allowed an essential increase in the yields. Apparently the P_2O_5–$POCl_3$ system absorbs water more effectively and facilitates the formation of ionic nitrilium intermediates that can promptly react with the aromatic ring. The reactions should be preferably carried out in toluene or without solvent.

We have investigated the possibility of reduction of dihydro-β-carbolines **VIII** to tetrahydro-β-carbolines and it is established that the application of $NaBH_4$ as a reducing agent gives the optimal results. Compound **IX** was obtained in 93% yield.

To transform the tetrahydroisoquinolines in the corresponding isoquinoline derivatives various reagents are used, e.g., *o*-(iodoxy)benzoic acid [29] or sulfur [30]. In dehydrogenation of compounds **VIII** the best result was obtained with palladium on carbon in toluene [31]; the yield of β-carboline derivative **X** was 94%.

Thus an approach has been developed to the synthesis of β-carboline derivatives containing 1,2,3-triazole ring with various substituents allowing to obtain series of compounds promising for further testing for biological activity.

EXPERIMENTAL

^{1}H NMR spectra of the synthesized compounds were recorded on a Bruker Avance 500 (500 MHz) spectrometer, solvent DMSO-d_6, internal reference TMS.

Aryl azides **IIIa–IIIe** were obtained by method [32] and were described in [24].

1-Aryl-5-methyl-1*H*-1,2,3-triazole-4-carboxylic acids and acyl chlorides **IVa–IVd** were obtained by procedures [33, 34] and were described in [24]**.**

***N*-[2-(1*H*-Indol-3-yl)ethyl]-5-methyl-1*H*-1,2,3-triazole-4-carboxamides VIIa–VIIf.** A slurry of 5 mmol of tryptamine hydrochloride **VIa–VIc** in 30 mL of dichlorethane was mixed with 1.38 g (10 mmol) of milled calcined K_2CO_3, a solution of 5 mmol of acid chloride **IVa–IVd** in 15 mL of CH_2Cl_2 was added dropwise at room temperature. The reaction mixture was stirred at 25°C for 12 h, then washed with water (3 × 20 mL). The organic layer was dried with $MgSO_4$, filtered, the solvent was evaporated, the residue was recrystallised from ethanol.

5-Methyl-*N*-[2-(6-methoxy-1*H*-indol-3-yl)ethyl]-1-phenyl-1*H*-1,2,3-triazole-4-carboxamide (VIIa). Yield 89%, mp 169–170°C . ^{1}H NMR spectrum, δ, ppm: 2.54 s (3H, Me), 2.93 t (2H, CH_2, *J* 6.0 Hz), 3.57 t (2H, CH_2, *J* 6.0 Hz), 3.76 s (3H, MeO), 6.67 d (1H, indole, *J* 8.3 Hz), 6.86 s (1H, indole), 7.07 s (1H, indole), 7.49 d (1H, indole, *J* 8.3 Hz), 7.60–7.70 br.s (5H, Ph), 8.66 br.s (1H, NH), 10.63 s (1H, NH). Found, %: C 67.26; H 5.42; N 18.56. $C_{21}H_{21}N_5O_2$. Calculated, %: C 67.18; H 5.64; N 18.65.

5-Methyl-1-phenyl-*N*-[2-(5-fluoro-1*H*-indol-3-yl) ethyl]-1*H*-1,2,3-triazole-4-carboxamide (VIIb). Yield 83%, mp 230–232°C. ^{1}H NMR spectrum, δ, ppm: 2.55 s (3H, Me), 3.02 t (2H, CH_2, *J* 7.0 Hz), 3.08 t (2H, CH_2, *J* 7.0 Hz), 6.92 t (1H, indole, *J* 8.8 Hz), 7.30–7.41 m (3H, Ar), 7.51–7.71 m (5H, Ar), 8.71 br.s (1H, NH), 11.17 s (1H, NH). Found, %: C 65.93; H 4.81; N 19.14. $C_{20}H_{18}FN_5O$. Calculated, %: C 66.10; H 4.99; N 19.27.

5-Methyl-1-phenyl-*N*-[2-(5-chloro-1*H*-indol-3-yl)-ethyl]-1*H*-1,2,3-triazole-4-carboxamide (VIIc). Yield 92%, mp 172–173°C. ^{1}H NMR spectrum, δ, ppm: 2.50 s (3H, Me), 2.92–3.00 m (2H, CH_2), 3.52–3.60 m (2H, CH_2), 7.07 d (1H, indole, *J* 8.4 Hz), 7.30 s (1H, indole), 7.36 d (1H, indole, *J* 8.4 Hz), 7.60–7.77 (Ar, 6H), 8.71 s (1H, NH), 11.07 s (1H, NH). Found, %: C 63.38; H 4.65; N 18.56. $C_{20}H_{18}ClN_5O$. Calculated, %: C 63.24; H 4.78; N 18.44.

5-Methyl-*N*-[2-(6-methoxy-1*H*-indol-3-yl)ethyl]-1-(4-fluorophenyl)-1*H*-1,2,3-triazole-4-carboxamide (VIId). Yield 78%, mp 227–228°C. ^{1}H NMR spectrum, δ, ppm: 2.55 s (3H, Me), 2.98 t (2H, CH_2, *J* 6.8 Hz), 3.07 t (2H, CH_2, *J* 6.8 Hz), 3.75 s (3H, MeO), 6.64 d (1H, indole, *J* 8.5 Hz), 6.87 s (1H, indole), 7.08 s (1H, indole), 7.41 d (1H, indole, *J* 8.5 Hz), 7.44–7.48 m (2H, C_6H_4), 7.62–7.67 m (2H, C_6H_4), 8.41 br.s (1H, NH), 10.80 s (1H, NH). Found, %: C 64.02; H 5.29; N 17.83. $C_{21}H_{20}FN_5O_2$. Calculated, %: C 64.11; H 5.12; N 17.80.

5-Methyl-1-(4-methoxyphenyl)-*N*-[2-(5-fluoro-1*H*-indol-3-yl)ethyl]-1*H*-1,2,3-triazole-4-carboxamide (VIIe). Yield 74%, mp 285–286°C. ^{1}H NMR spectrum, δ, ppm: 2.55 s (3H, Me), 3.02 br.s (2H, CH_2), 3.09 br.s (2H, CH_2), 3.85 s (3H, MeO), 6.91 t (1H, indole, *J* 8.8 Hz), 7.13 d (2H, C_6H_4, *J* 7.2 Hz), 7.31–7.39 m (3H, indole), 7.48 d (2H, C_6H_4, *J* 7.2 Hz), 8.61 br.s (1H, NH), 11.21 br.s (1H, NH). Found, %: C 64.21; H 5.25; N 17.71. $C_{21}H_{20}FN_5O_2$. Calculated, %: C 64.11; H 5.12; N 17.80.

5-Methyl-1-(4-methylphenyl)-*N*-[2-(5-fluoro-1*H*-indol-3-yl)ethyl]-1*H*-1,2,3-triazole-4-carboxamide (VIIf). Yield 91%, mp 188–189°C. ^{1}H NMR spectrum, δ, ppm: 2.42 s (3H, Me), 2.55 s (3H, Me), 2.96 t (2H, CH_2, *J* 7.2 Hz), 3.56 t (2H, CH_2, *J* 7.2 Hz), 7.07 d (1H,

indole, *J* 8.0 Hz), 7.31 s (1H, indole), 7.37 d (1H, indole, *J* 8.0 Hz), 7.44 d (2H, C_6H_4, *J* 7.8 Hz), 7.50 d (2H, C_6H_4, *J* 7.8 Hz), 7.67 s (1H, indole), 8.69 t (1H, NH, *J* 5.4 Hz), 11.11 br.s (1H, NH). Found, %: C 64.22; H 5.27; N 17.63. $C_{21}H_{20}ClN_5O$. Calculated, %: C 64.04; H 5.12; N 17.78.

1-(1*H*-1,2,3-Triazol-4-yl)-4,9-dihydro-3*H*-β-carboline derivatives VIIIa–VIIIf. In 25 mL of anhydrous toluene was dissolved 0.88 mmol of amide **VII** and 1.24 g (8.9 mmol) of P_2O_5 was dispersed**,** 0.83 mL (8.9 mmol) $POCl_3$ was added dropwise. After adding $POCl_3$ the reaction mixture was heated for 12 h, the solvent and $POCl_3$ were distillated in a vacuum, then cooled and mixed with ~25 g of crushed ice. The resulting mixture was neutralised with 20% solution of NaOH, the reaction product was extracted with toluene. The extract was dried with $MgSO_4$, the solvent was evaporated, the residue was recrystallised from alcohol.

1-(5-Methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)-7-methoxy-4,9-dihydro-3*H*-β-carboline (VIIIa). Yield 81%, mp 190–191°C. ^{1}H NMR spectrum, δ, ppm: 2.63s (3H, Me), 2.89 t (2H, CH_2, *J* 7.6 Hz), 3.79 s (3H, MeO), 4.10 t (2H, CH_2, *J* 7.6 Hz), 6.79 d (1H, carboline, *J* 8.4 Hz), 7.21 s (1H, carboline), 7.54 d (1H, carboline, *J* 8.4 Hz), 7.64–7.73 m (5H, Ph), 11.01 s (1H, NH). Found, %: C 70.45; H 5.48; N 19.76. $C_{21}H_{19}N_5O$. Calculated, %: C 70.57; H 5.36; N 19.59.

1-(5-Methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)-6-fluoro-4,9-dihydro-3*H*-β-carboline (VIIIb). Yield 74%, mp 152–153°C. ^{1}H NMR spectrum, δ, ppm: 2.63 s (2H, Me), 2.81 t (2H, CH_2, *J* 6.8 Hz), 3.29 t (2H, CH_2, *J* 6.8 Hz), 7.00 t (1H, carboline, *J* 8.8 Hz), 7.58–7.79 m (7H, Ar), 11.34 s (1H, NH). Found, %: C 69.75; H 4.82; N 20.16. $C_{20}H_{16}FN_5$. Calculated, %: C 69.55; H 4.67; N 20.28.

1-(5-Methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)-6-chloro-4,9-dihydro-3*H*-β-carboline (VIIIc). Yield 89%, mp 203–205°C. ^{1}H NMR spectrum, δ, ppm: 2.65 s (3H, Me), 2.89 t (2H, CH_2, *J* 7.6 Hz), 4.02 t (2H, CH_2, *J* 7.6 Hz), 7.21 d (1H, carboline, *J* 8.4 Hz), 7.57–7.67 m (6H, Ar), 7.96 s (1H, carboline), 11.36 s (1H, NH). Found, %: C 66.44; H 4.54; N 19.31. $C_{20}H_{16}ClN_5$. Calculated, %: C 66.39; H 4.46; N 19.36.

1-[5-Methyl-1-(4-phluorophenyl)-1*H*-1,2,3-triazol-4-yl]-7-methoxy-4,9-dihydro-3*H*-β-carboline (VIIId). Yield 68%, mp 185–186°C. ^{1}H NMR spectrum, δ, ppm: 2.65 s (3H), 2.87 t (2H, CH_2, *J* 7.2 Hz), 3.26 t (2H, CH_2, *J* 7.2 Hz), 3.77 s (3H, MeO), 6.74 d (1H, carboline, *J* 8.6 Hz), 7.25 s (1H, carboline), 7.48 t (2H, C_6H_4, *J* 8.6 Hz), 7.59 d (1H, carboline, *J* 8.6 Hz), 7.62–7.67 m (2H, C_6H_4), 11.17 s (1H, NH). Found, %: C 67.27; H 4.74; N 18.76. $C_{21}H_{18}FN_5O$. Calculated, %: C 67.19; H 4.83; N 18.66.

1-[5-Methyl-1-(4-metoxyphenyl)-1*H*-1,2,3-triazol-4-yl]-6-fluoro-4,9-dihydro-3*H*-β-carboline (VIIIe). Yield 75%, mp 182–184°C. ^{1}H NMR spectrum, δ, ppm: 2.67 s (2H, Me), 2.91 br.s (2H, CH_2), 3.26 t (2H, CH_2, *J* 7.2 Hz), 3.92 s (3H, MeO), 7.01 t (1H, carboline, *J* 8.8 Hz), 7.13 d (2H, C_6H_4, *J* 7.4 Hz), 7.52 d (2H, C_6H_4, *J* 7.4 Hz), 7.54–7.62 m (2H, carboline), 11.33 s (1H, NH). Found, %: C 67.29; H 4.74; N 18.78. $C_{21}H_{18}FN_5O$. Calculated, %: C 67.19; H 4.83; N 18.66.

1-[5-Methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazol-4-yl]-6-chloro-4,9-dihydro-3*H*-β-carboline (VIIIf). Yield 81%, mp 211–213°C. ^{1}H NMR spectrum, δ, ppm: 2.46 s (3H, Me), 2.60 s (3H, Me), 2.94 t (2H, CH_2, *J* 8.2 Hz), 4.01 t (2H, CH_2, *J* 8.2 Hz), 7.24 d (1H, carboline, *J* 8.7 Hz), 7.49 d (2H, C_6H_4, *J* 8.3 Hz), 7.57 d (2H, C_6H_4, *J* 8.3 Hz), 7.71 d (1H, carboline, *J* 8.7 Hz), 7.72 s (1H, carboline), 11.55 s (1H, NH). Found, %: C 67.01; H 4.74; N 18.81. $C_{21}H_{18}ClN_5$. Calculated, %: C 67.11; H 4.83; N 18.63.

1-(5-Methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)-6-chloro-2,3,4,9-tetrahydro-1*H*-β-carboline (IX). To a solution of **3**62 mg (1 mmol) of compound **VIIIc** in 15 mL of methanol cooled in the ice bath was added 75 mg of $NaBH_4$. The mixture was stirred at room temperature for 5 min, then boiled for 1 h. Methanol was evaporated, the residue was dissolved in CH_2Cl_2 and washed with 10% NaOH, methylene chloride was evaporated. Yield 91%, mp 191–192°C. ^{1}H NMR spectrum, δ, ppm: 2.55 s (3H, Me), 2.81 m (2H, CH_2), 3.62 m (2H, CH_2), 4.12 br.s (1H, CH), 5.45 br.s (1H, NH), 7.14 d (1H, carboline, *J* 8.4 Hz), 7.53 s (1H, carboline), 7.55–7.69 m (6H, Ar), 11.05 s (1H, NH). Found, %: C 66.15; H 4.83; N 19.41. $C_{20}H_{18}ClN_5$. Calculated, %: C 66.02; H 4.99; N 19.25.

1-[5-Methyl-1-(4-methylphenyl)-6-chloro-1*H*-1,2,3-triazol-4-yl]-9*H*-β-carboline (X). 376 mg (1 mmol) of the compound **VIIIf** and 100 mg of 5% Pd/C were mixed. The mixture was ground to powder-like condition and heated at 150°C for 30 min. To the reaction mixture 100 mL of CH_2Cl_2 was added, the mixture was stirred and filtered. The solvent was distilled off, the residue was recrystallised from a mixture alcohol-DMF. Yield 84%, mp >250°C. ^{1}H NMR spectrum, δ, ppm: 2.44 s (3H, Me), 2.65 s (3H, Me), 7.30 d (1H, carboline, *J* 8.0 Hz), 7.53 d (2H, C_6H_4, *J* 8.4 Hz), 7.67 d (2H, C_6H_4, *J* 8.4 Hz), 7.73–7.78 m (2H, carboline), 8.01 d (1H,

carboline, *J* 8.0 Hz), 8.54 d (1H, carboline, *J* 5.1 Hz), 12.05 s (1H, NH). Found, %: C 67.38; H 4.41; N 18.61. $C_{21}H_{16}ClN_5$. Calculated, %: C 67.47; H 4.31; N 18.73.

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