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Synthesis and Dehydrogenation of Spinaceamine and Spinacine 4-Hetaryl Derivatives

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Abstract—The reaction of histamine and histidine with various hetarylaldehydes under the conditions of the base-catalyzed Pictet–Spengler process affords 4-hetaryl-substituted derivatives of spinaceamine and spinacine. The dehydrogenation of the 4-hetaryl-substituted spinaceamine derivatives using elemental sulfur in DMF at 120–130°C led to the formation of 4-hetaryl derivatives of imidazo[4,5-*c*]-pyridine. Under similar conditions the 4-hetaryl-substituted spinacine derivatives suffered both the dehydrogenation and oxidative decarboxylation resulting in the products identical to the compounds obtained by the dehydrogenation of 4-hetaryl-substituted spinaceamine.

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Formerly we described by an example of 4-aryl-substituted spinaceamine and spinacine their dehydrogenation by elemental sulfur in DMF at 120–150°C. The dehydrogenation of 4-aryl-substituted spinaceamine provided 4-aryl derivatives of imidazo-[4,5-*c*]pyridine, and the 4-aryl-substituted spinacine under the same conditions underwent both aromatization and oxidative decarboxylation giving products identical to those obtained by dehydrogenation of the 4-aryl derivatives of spinaceamine [1].

In extension of the study of these processes in the series of spinaceamine derivatives we performed the dehydrogenation of 4-hetaryl-substituted spinaceamines and spinacines aiming at a development of a synthesis of imidazo[4,5-*c*]pyridine 4-hetaryl derivatives that were difficultly available and might be interesting as promising synthons for the preparation of biologically active substances.

The reaction of histamine (**I**) and histidine (**II**) with various hetarylaldehydes afforded new hetaryl-substituted spinaceamines **IIIa–IIIi** and spinacines **IVa–IVk** containing in the position *4* of the tetrahydropyridine ring pyridine, thiophene, quinoline, pyrazole, furan, pyrrole, indole, and benzimidazole fragments (see the scheme).

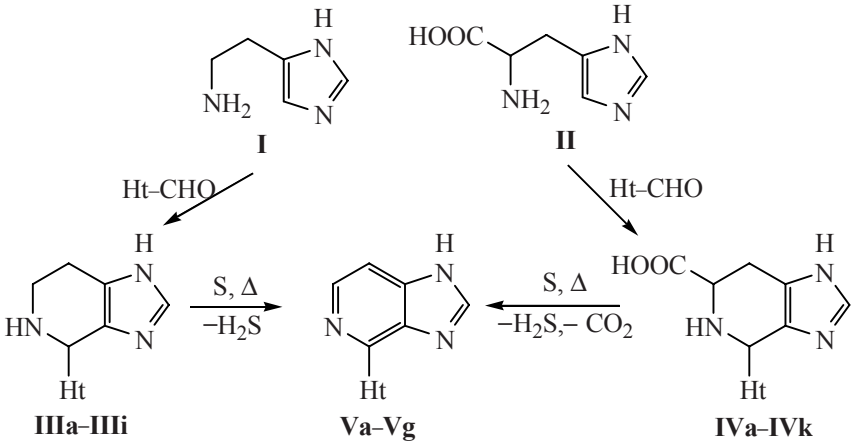
4-Hetaryl-substituted spinaceamines **IIIa–IIIi** and spinacines **IVa–IVk** whose yields attained respectively 33–88 and 30–85% are colorless substances purified by crystallization from water. Their composition and structure were proved by elemental analysis and ^{1}H NMR spectra.

The heating of 4-hetarylspinaceamines **IIIa–IIIg** in DMF with elemental sulfur at 120–130°C for 2–7 h (till the end of hydrogen sulfide evolution) resulted in 4-hetaryl-substituted imidazo-[4,5-*c*]pyridines **Va–Vg** in 51–61% yields. In the ^{1}H NMR spectra of these compounds alongside the signals of the imidazole proton H^2 (7.81–8.78 ppm) and of the protons from the hetaryl fragment signals were present from protons H^6 (7.87–9.89) and H^7 (7.33–8.61 ppm) of the pyridine ring with a coupling constant of 5.3–8.0 Hz.

In contrast to spinaceamines **IIIa–IIIg** compounds **IIIh** and **IIIi** containing in the position *4* of the tetrahydropyridine ring pyrrole and furan residues respectively in the course of the dehydrogenation under the chosen conditions suffered decomposition affording tarry products whose structure we failed to establish.

At heating 4-hetarylspinacines **IVa–IVg** under analogous conditions they underwent both aromatization and oxidative decarboxylation giving products identical

Scheme.



Ht = 3-pyridyl (**IIIa–Va**), 4-pyridyl (**IIIb–Vb**), 2-thienyl (**IIIc–Vc**), quinolin-8-yl (**IIId–Vd**), 1,3-dimethyl-2-oxobenzimidazol-5-yl (**IIIe–Ve**), 1-phenyl-3-methylpyrazol-4-yl (**IIIf–Vf**), 1,3-diphenylpyrazol-4-yl (**IIIg–Vg**), N-methylpyrrol-2-yl (**IIIh**), 5-methyl-2-furyl (**IIIi**), 1,3,5-trimethylpyrazol-4-yl (**IVh**), N-methylpyrrol-2-yl (**IVi**), N-methylindol-3-yl (**IVj**), N-benzylindol-3-yl (**IVk**).

to compounds **Va–Vg** obtained previously by dehydrogenation of spinaceamines **IIIa–IIIg**. 4-Hetaryl-substituted imidazo[4,5-*c*]pyridines **Va–Vg** formed at the dehydrogenation with sulfur of spinaceamines **IIIa–IIIg** and spinacines **IVa–IVg** have similar melting points and ^{1}H NMR spectra.

The attempts at the dehydrogenation under similar conditions of spinacines **IVh–IVk** containing in the position *4* of the tetrahydropyridine ring 1,3,5-trimethylpyrazole, pyrrole, and indole fragments led to tarry reaction products whose structure we failed to establish.

Hence the results obtained suggest a conclusion that a new simple method was developed for the synthesis of 4-hetarylimidazo[4,5-*c*]pyridines **Va–Vg** by the dehydrogenation of 4-hetaryl-substituted spinaceamines **IIIa–IIIg** or spinacines **IVa–IVg** with elemental sulfur in DMF at 120–130°C.

The prepared new derivatives of spinaceamine **IIIa–IIIi**, spinacine **IVa–IVk**, and imidazo[4,5-*c*]pyridine **Va–Vg** may be interesting for pharmacology since among analogous compounds of this series substances have been revealed exhibiting a wide range of biological action [2].

EXPERIMENTAL

^{1}H NMR spectra of compounds synthesized were registered on a spectrometer Varian Gemini-200 at operating frequency 200 MHz, internal reference HMDS. The purity and homogeneity of the products obtained was checked by TLC on Silufol UV-254 plates (eluents ethanol, chloroform; spots visualization by iodine vapor or under UV irradiation).

Spinaceamines **IIIb** and **IIIc** were obtained by procedures [3] and [4] respectively. Spinacine **IVb** was obtained as described in [5].

Spinaceamine 4-hetaryl derivatives IIIa, IIId – IIIi. To a solution of 10 mmol of histamine dihydrochloride (**I**) in 20 ml of water were added solutions of 30 mmol of NaOH in 10 ml of water and 10 mmol of an appropriate aldehyde in 40 ml of ethanol. The reaction mixture was heated for 5–7 h on a boiling water bath. On cooling the separated pecipitate was filtered off and recrystallized from water.

4-(3-Pyridyl)spinaceamine (IIIa). Yield 1.46 g (73%), mp 171–173°C. ^{1}H NMR spectrum CD_3OD), δ, ppm: 3.32 t (2H, 7-CH_2), 3.62 t (2H, 6-CH_2), 5.90 s (1H, H*4*), 7.38–7.73 m (1H, H*5′*), 7.85 s (1H, H*2*), 8.11 d (1H, H*4′*, *J* 7.2 Hz), 8.55 d (1H, H*6′*, *J* 7.2 Hz), 8.88 s (1H, H*2′*). Found, %: C 65.93; H 6.07; N 27.80. $C_{11}H_{12}N_4$. Calculated, %: C 65.98; H 6.04; N 27.98.

4-(Quinolin-8-yl)spinaceamine (IIId). Yield 1.2 g (48%), mp 97–99°C. ^{1}H NMR spectrum (CD_3OD), δ, ppm: 2.73 t (2H, 7-CH_2), 2.99 t (2H, 6-CH_2), 6.19 s (1H, H*4*), 7.24 d (1H, H*7′*, *J* 7.2 Hz), 7.40–7.53 m (3H, H*3′,5′,6′*), 7.79 d (1H, H*4′*, *J* 7.2 Hz), 8.09 d (1H, H*2′*, *J* 7.2 Hz), 8.80s (1H, H*2*). Found, %: C 71.82; H 5.67; N 22.19. $C_{15}H_{14}N_4$. Calculated, %: C 71.98; H 5.64; N 22.39.

4-(1,3-Dimethyl-2-oxobenzimidazol-5-yl)spinaceamine (IIIe). Yield 1.3 g (46%), mp 145–147°C. ^{1}H NMR spectrum (CD_3OD), δ, ppm: 2.77 t (2H, 7-CH_2), 3.10 t (2H, 6-CH_2), 3.31–3.35 m (6H, 2CH_3), 5.08 s (1H, H^4), 7.00–7.06 m (3H, H$^{4',6',7'}$), 7.51 s (1H, H^2). Found, %: C 63.54; H 6.07; N 24.60. $C_{15}H_{17}N_5O$. Calculated, %: C 63.58; H 6.05; N.72.

4-(3-Methyl-1-phenylpyrazol-4-yl)spinaceamine (IIIf). Yield 2.45 g (88%), mp 198–200°C. ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 2.34 s (3H, CH_3), 3.20 t (2H, 7-CH_2), 3.73 t (2H, 6-CH_2), 6.03 s (1H, H^4), 7.26–7.64 m (5H, C_6H_5), 8.15 s (1H, H^2), 8.68 s (1H, H$^{5'}$). Found, %: C 68.68; H 6.18; N 25.00. $C_{16}H_{17}N_5$. Calculated, %: C 68.79; H.14; N 25.07.

4-(1,3-Diphenylpyrazol-4-yl)spinaceamine (IIIg). Yield 2.39 g (70%), mp 253–255°C. ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 3.19 t (2H, 7-CH_2), 3.74 t (2H, 6-CH_2), 6.22 s (1H, H^4), 7.30–7.78 m (10H, 2C_6H_5), 8.37 s (1H, H$^{5'}$), 8.69 s (1H, H^2). Found, %: C.79; H 5.66; N 20.39. $C_{21}H_{19}N_5$. Calculated, %: C 73.88; H 5.61; N 20.51.

4-(N-Methylpyrrol-2-yl)spinaceamine (IIIh). Yield 0.67 g (33%), mp 148–150°C. ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 3.21 s (2H, 7-CH_2), 3.67 s (2H, 6-CH_2), 3.75 s (3H, CH_3), 5.99 s (1H, H^4), 6.05–6.78 m (3H, H$^{3'-5'}$), 8.73 s (1H, H^2). Found, %: C 65.25; H.06; N 27.59. $C_{11}H_{14}N_4$. Calculated, %: C 65.32; H 6.98; N 27.70.

4-(5-Methyl-2-furyl)spinaceamine (IIIi). Yield 1.22 g (60%), mp 87–88°C. ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 2.22 s (3H, CH_3), 3.18 s (2H, 7-CH_2), 3.67 s (2H, 6-CH_2), 6.02 s (1H, H^4), 6.08 s (1H, H$^{4'}$), 6.41 s (1H, H$^{3'}$), 8.64 s (1H, H^2). Found, %: C.83; H 6.51; N 20.51. $C_{11}H_{14}N_4$. Calculated, %: C 65.00; H 6.45; N 20.68.

Spinacine 4-hetaryl derivatives IVa, IVc–IVk. To a solution of 10 mmol L-hustidine hydrochloride monohydrate (**II**) in 20 ml of water were added solutions of 30 mmol of NaOH in 10 ml of water and 10 mmol of an appropriate aldehyde in 40 ml of ethanol. The reaction mixture was heated for 5–7 h on a boiling water bath. Ethanol was distilled off in a vacuum, the residue was cooled and acidified with 6N HCl till pH 4–5. The separated pecipitate was filtered off and recrystallized from water.

4-(3-Pyridyl)spinacine (IVa). Yield 1.24 g (51%), mp 228–230°C (decomp.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.98 q (2H, 7-CH_2), 3.85 t (1H, H^6), 5.31 s (1H, H^4), 7.46 q (1H, H$^{5'}$), 7.58 s (1H, H$^{2'}$), 7.80 d (1H, H$^{4'}$, *J* 8.1 Hz), 8.61 d (1H, H$^{6'}$, *J* 8.1 Hz), 8.66 s (1H, H^2). Found, %: C 58.97; H 4.99; N 22.91. $C_{12}H_{12}N_4O_2$. Calculated, %: C 59.01; H.95; N 22.93.

4-(2-Thienyl)spinacine (IVc). Yield 2.12 g (85%), mp 200–202°C. ^{1}H NMR spectrum (CD_3OD), δ, ppm: 3.12 q (2H, 7-CH_2), 4.08 q (1H, H^6), 5.96 s (1H, H^4), 7.09 d (1H, H$^{3'}$, *J* 5.0 Hz), 7.28 q (1H, H$^{4'}$), 7.54 d (1H, H$^{5'}$, *J* 5.0 Hz), 7.81 s (1H, H^2). Found, %: C 52.89; H 4.48; N 16.78; S 12.81. $C_{11}H_{11}N_3O_2S$. Calculated, %: C 53.00; H 4.45; N 16.86; S 12.86.

4-(Quinolin-8-yl)spinacine (IVd). Yield 1.06 g (36%), mp 178–180°C. ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 3.66 q (2H, 7-CH_2), 4.41 d (1H, H^6), 6.69 s (1H, H^4), 7.55–7.73 m (1H, H$^{6'}$), 8.09 d (1H, H$^{7'}$, *J* 8.1 Hz), 8.15 d (1H, H$^{5'}$, *J* 8.1 Hz), 8.22 t (1H, H$^{3'}$), 8.44 d (1H, H$^{4'}$, *J* 8.1 Hz), 8.64 s (1H, H^2), 8.88 d (1H, H$^{2'}$, *J* 8.1 Hz). Found, %: C 65.19; H 4.80; N 18.90. $C_{16}H_{14}N_4O_2$. Calculated, %: C 65.30; H 4.79; N 19.04.

4-(1,3-Dimethyl-2-oxobenzimidazol-5-yl)-spinacine (IVe). Yield 2.62 g (80%), mp 253–255°C (decomp.). ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 3.40 s (3H, CH_3), 3.46 s (3H, CH_3), 3.53 q (2H, 7-CH_2), 3.76 t (1H, H^6), 4.36 s (1H, H^4), 7.26 d (1H, H$^{6'}$, *J* 8.1 Hz), 7.57 d (1H, H$^{4'}$), 7.75 d (1H, H$^{7'}$, *J* 8.1 Hz), 8.78 s (1H, H^2). Found, %: C 58.59; H 5.21; N 21.32. $C_{16}H_{17}N_5O_3$. Calculated, %: C 58.70; H 5.24; N 21.40.

4-(3-Methyl-1-phenylpyrazol-4-yl)spinacine (IVf). Yield 1.62 g (50%), mp 227–229°C (decomp.). ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 2.39 s (3H, CH_3), 3.44 q (2H, 7-CH_2), 4.61 t (1H, H^6), 6.25 s (1H, H^4), 7.26–7.67 m (5H, C_6H_5), 8.14 s (1H, H^2), 8.69 s (1H, H$^{5'}$). Found, %: C 62.99; H 5.33; N 21.39. $C_{17}H_{17}N_5O_2$. Calculated, %: C 63.14; H 5.30; N 21.66.

4-(1,3-Diphenylpyrazol-4-yl)spinacine (IVg). Yield 2.46 g (64%), mp 205–208°C (decomp.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.95 q (2H, 7-CH_2), 3.97 t (1H, H^6), 5.17 s (1H, H^4), 7.28–7.90 m (10H, 2C_6H_5), 8.14 s (1H, H$^{5'}$), 8.57 s (1H, H^2). Found, %: C.54; H 5.00; N 18.02. $C_{22}H_{19}N_5O_2$. Calculated, %: C 68.56; H 4.97; N 18.17.

4-(1,3,5-Trimethylpyrazol-4-yl)spinacine (IVh). Yield 1.9 g (70%), mp 211–213°C (decomp.). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.83 s (3H, CH_3), 1.97 C (3H, CH_3), 3.10 q (2H, 7-CH_2), 3.60 s (3H, CH_3), 4.10 d (1H, H^6), 5.44 s (1H, H^4), 7.85 s (1H, H^2). Found, %: C 56.64; H 6.30; N 25.42. $C_{13}H_{17}N_5O_2$. Calculated, %: C 56.71; H 6.23; N.44.

4-(*N*-Methylpyrrol-2-yl)spinacine (IVi). Yield 0.74 g (30%), mp 215–217°C (decomp.). ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 3.44 q (2H, 7-CH_2), 3.77 s (3H, CH_3), 4.45 d (1H, H^6), 5.98 s (1H, H^4), 6.04–6.78 m (3H, H$^{3'-5'}$), 8.71 s (1H, H^2). Found, %: C 58.50; H.71; N 22.66. $C_{12}H_{14}N_4O_2$. Calculated, %: C 58.52; H 5.73; N 22.75.

4-(*N*-Methylindol-3-yl)spinacine (IVj). Yield 0.98 g (33%), mp 240–242°C (decomp.). ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 3.62 q (2H, 7-CH_2), 3.85 s (3H, CH_3), 3.93 d (1H, H^6), 4.33 s (1H, H^4), 6.77–7.49 m (4H, C_6H_4), 7.55 s (1H, H$^{2'}$), 8.71 C (1H, H^2). Found, %: C 64.80; H 5.47; N 18.82. $C_{16}H_{16}N_4O_2$. ВьчθС-λενО, %: C 64.85; H 5.44; N.91.

4-(*N*-Benzylindol-3-yl)spinacine (IVk). Yield 2.6 g (70%), mp 200–202°C (decomp.). ^{1}H NMR spectrum (CD_3COOD), δ, ppm: 3.55 q (2H, 7-CH_2), 4.58 t (1H, H^6), 5.34 s (2H, $CH_2C_6H_5$), 6.45 s (1H, H^4), 7.05–7.57 m (9H, C_6H_4, $CH_2C_6H_5$), 8.50 s (1H, H$^{2'}$), 8.74 s (1H, H^2). Found, %: C 70.89; H 5.50; N 14.96. $C_{22}H_{20}N_4O_2$. Calculated, %: C 70.95; H5.41; N 15.05.

Imidazo[4,5-*c*]pyridine 4-hetaryl derivatives Va–Vg. *a.* A mixture of 10 mmol of spinaceamine **IIIa**–**IIIg** in 50 ml of DMF and 0.7 g (22 mmol) of elemental sulfur was heated for 2–7 h at 120–130°C till the end of H_2S evolution. Then DMF was distilled off in a vacuum till dryness, and the residue was recrystallized from water.

b. To a solution of 10 mmol of spinacine **IVa–IVg** in 50 ml of DMF was added 0.7 g (22 mmol) of elemental sulfur. The mixture was treated as in procedure *a*.

4-(3-Pyridyl)imidazo[4,5-*c*]pyridine (Va). *a.* Yield 1.0 g (51%), mp 252–254°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.63 t (1H, H$^{5'}$), 7.69 d (1H, H^7, *J* 5.6 Hz), 8.54 d (1H, H^6, *J* 5.6 Hz), 8.55 s (1H, H^2), 8.72 d (1H, H$^{4'}$, *J* 7.2 Hz), 9.07 d (1H, H$^{6'}$, *J* 7.2 Hz), 9.94 C (1H, H$^{2'}$). Found, %: C 67.26; H 4.12; N 28.43. $C_{11}H_8N_4$. Calculated, %: C 67.33; H 4.11; N 28.56.

b. Yield 1.3 g (67%), mp 253–255°C. Found, %: C 67.24; H 4.13; N 28.47.

4-(4-Pyridyl)imidazo[4,5-*c*]pyridine (Vb). *a*. Yield 1.18 g (60%), mp 273–275°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.60 d (1H, H^7, *J* 5.3 Hz), 8.43 d (1H, H^6, *J*5.3Hz), 8.47 s (1H, H^2), 8.64 d (2H, H$^{2',6'}$, *J* 4.5 Hz), 8.65 d (2H, H$^{3',5'}$, *J* 4.5 Hz). Found, %: C 67.29; H 4.15; N 28.49. $C_{11}H_8N_4$. Calculated, %: C 67.33; H 4.11; N 28.56.

b. Yield 1.2 g (61%), mp 273–275°C. Found, %: C 67.23; H 4.13; N 28.49.

4-(2-Thienyl)imidazo[4,5-*c*]pyridine (Vc). *a.* Yield 1.11 g (55%), mp 198–200°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.36 t (1H, H$^{4'}$), 7.86 d (1H, H^7, *J* 5.5 Hz), 8.03d (1H, H$^{3'}$, *J* 4.6 Hz), 8.30 d (1H, H^6, *J* 5.5 Hz), 8.41 d (1H, H$^{5'}$, *J* 4.6 Hz), 8.67 s (1H, H^2). Found, %: C 59.63; H 3.52; N 20.79; S 15.78. $C_{10}H_7N_3S$. Calculated, %: C 59.68; H 3.51; N 20.88; S 15.93.

b. Yield 1.76 g (88%), mp 197–199°C. Found, %: C 59.59; H 3.53; N 20.74; S 15.82.

4-(Quinolin-8-yl)imidazo[4,5-*c*]pyridine (Vd). *a.* Yield 1.5 g (60%), mp >300°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.71–7.84 m (1H, H$^{6'}$), 7.97 d (1H, H$^{7'}$, *J*.5Hz), 8.20–8.27 m (2H, H$^{4',5'}$), 8.55 d (1H, H^7, *J* 7.6 Hz), 8.76 s (1H, H^2), 8.75–9.15 m (2H, H$^{2',3'}$), 9.86 d (1H, H^6, *J* 7.6 Hz). Found, %: C 73.02; H 4.15; N 22.67. $C_{15}H_{10}N_4$. Calculated, %: C 73.15; H 4.09; N 22.75.

b. Yield 1.70 g (69%), mp > 300°C. Found, %: C 73.12; H 4.13; N.67.

4-(1,3-Dimethyl-2-oxobenzimidazol-5-yl)-imidazo[4,5-*c*]pyridine (Ve). *a.* Yield 1.70 g (60%), mp 226–228°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.33 s (6H, 2CH_3), 7.30 d (1H, H^7, *J* 8.0 Hz), 7.84 d (1H, H^6, *J* 8.0 Hz), 8.35–8.39 m (3H, C_6H_3), 8.78 s (1H, H^2). Found, %: C 64.43; H 4.72; N 24.97. $C_{15}H_{13}N_5O$. Calculated, %: C 64.50; H.69; N 25.08.

b. Yield 1.67 g (63%), mp 226–228°C. Found, %: C 64.41; H 4.72; N 24.98.

4-(3-Methyl-1-phenylpyrazol-4-yl)imidazo[4,5-*c*]-pyridine (Vf). *a.* Yield 1.68 g (61%), mp 103–105°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.68 s (3H, CH_3), 7.20–7.80 m (6H, C_6H_5, H^2), 8.20–8.42 m (2H, H^7, H$^{5'}$), 9.33 d (1H, H^6). Found, %: C 69.68; H 4.79; N.37. $C_{16}H_{13}N_5$. Calculated, %: C 69.80; H 4.76; N 25.44.

b. Yield 1.62 g (57%), mp 102–103°C. Found, %: C.79; H 4.81; N 25.33.

4-(1,3-Diphenylpyrazol-4-yl)imidazo[4,5-*c*]-pyridine (Vg). *a.* Yield 2.06 g (63%), mp 175–177°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.30–7.75 m (10H, 2C_6H_5), 7.82–8.00 m (2H, H^2, H$^{5'}$), 8.35 d (1H, H^7, *J* 7.5 Hz), 9.17 d (1H, H^6, *J* 7.5 Hz). Found, %: C 74.69; H 4.55; N 20.65. $C_{21}H_{15}N_5$. Calculated, %: C 74.75; H 4.48; N 20.76.

b. Yield 1.11 g (33%), mp 175–177°C. Found, %: C 74.66; H 4.52; N 20.66.

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