Synthesis of derivatives of a new heterocyclic system pyrazolo[3,4-*b*]pyrido[1′,2′:1,2]imidazo[4,5-*d*]pyridine

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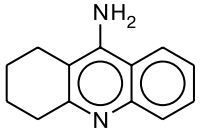
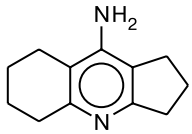
1-[4-Aminoarylpyrazolo[3,4-*b*]pyridin-5-yl]pyridinium chlorides undergo cyclization under reflux in *tert*-butanol in the presence of an excess of potassium *tert*-butoxide to form tetracyclic derivatives of pyrazolo[3,4-*b*]pyrido[1′,2′:1,2]imidazo[4,5-*d*]pyridine. The reaction scheme of the processes is proposed. The structures of the reaction products were confirmed by physicochemical methods.

Key words: 2-chloroacetamido-4-cyanopyrazole, 1-[4-aminopyrazolo[3,4-*b*]pyridin-5-yl]pyridinium chloride, pyrazolo[3,4-*b*]pyrido[1′,2′:1,2]imidazo[4,5-*d*]pyridine, 4,5-diaminopyrazolo[3,4-*b*]pyridine.

In recent years, it has been found[1—3] that agents activating the congitive functions, for example, anticholinesterase drugs, such as tacrine and amiridine (4-aminopyridine derivatives), can be used in the treatment of various asthenic states, central nervous system disorders, and intellectual amnestic disorders, which cannot be presently effectively treated by therapeutic agents.



Tacrine



Amiridine

These drugs are used in the treatment of various dementias, although primarily as auxiliary pharmaceuticals. Hence, there is a strong need to design new effective pharmaceuticals, which can provide approaches to the treatment of the above-mentioned diseases and to design new drugs, which can improve memory. The design of compounds serving as activators of the congitive functions is one of the main ways of struggling against these diseases.

In this connection, investigations of approaches to the synthesis of compounds containing the 4-aminopyridine fragment hold promise in designing new pharmaceuticals for the treatment of neurodegenerative diseases, for example, of Alzheimer's disease.[4]

The aim of the present study was to develop a procedure for the synthesis of such compounds of the aminopyrazolopyridine series.

Alkylation of 3-amino-4-cyanopyrazole (**1**)[5] with *o*-chlorobenzyl chloride in methanol in the presence of potassium carbonate afforded a mixture of isomeric pyrazole derivatives, *viz.*, 3-amino-1-(2-chlorobenzyl)-4-cyanopyrazole (**2**) and 5-amino-1-(2-chlorobenzyl)-4-cyanopyrazole (**3**) (Scheme 1). Pyrazole **2** was isolated in individual state in 57% yield after double crystallization from ethanol. The ^{1}H NMR spectrum of **2** (DMSO-d_6, δ) shows signals at 5.20 (s, 2 H, N(1)CH_2), 5.64 (br.s, 2 H, NH_2), 7.13—7.49 (m, 4 H, C_6H_4), and 8.24 (s, 1 H, H(5)). The structure of compound **2** was established based on the NOEDIFF spectrum. In the case of saturation of the signal of the methylene group (δ 5.20), a response for H(5) was observed, which indicates that the proton at position 5 of the pyrazole ring is in the proximity to the methylene group. An increase in the intensity of the signal for H(5) due to NOE was 12%. The assignment of some signals of the 5-NH_2 isomer **3** in the isomeric mixture of compounds **2** and **3** (for example, the signal at δ 7.64 (s, 1 H, H(3)) was made based on the comparison of the ^{1}H NMR spectra of compound **2** and its phenyl-substituted analog **8**[6] (DMSO-d_6, δ: 6.55 (br.s, 2 H, NH_2); 7.40—7.55 (m, 5 H, Ph); 7.76 (s, 1 H, H(3)), which can be considered as a model of the 5-NH_2 isomer, and the ratio of pyrazole **2** to **3** was estimated at 4 : 1.

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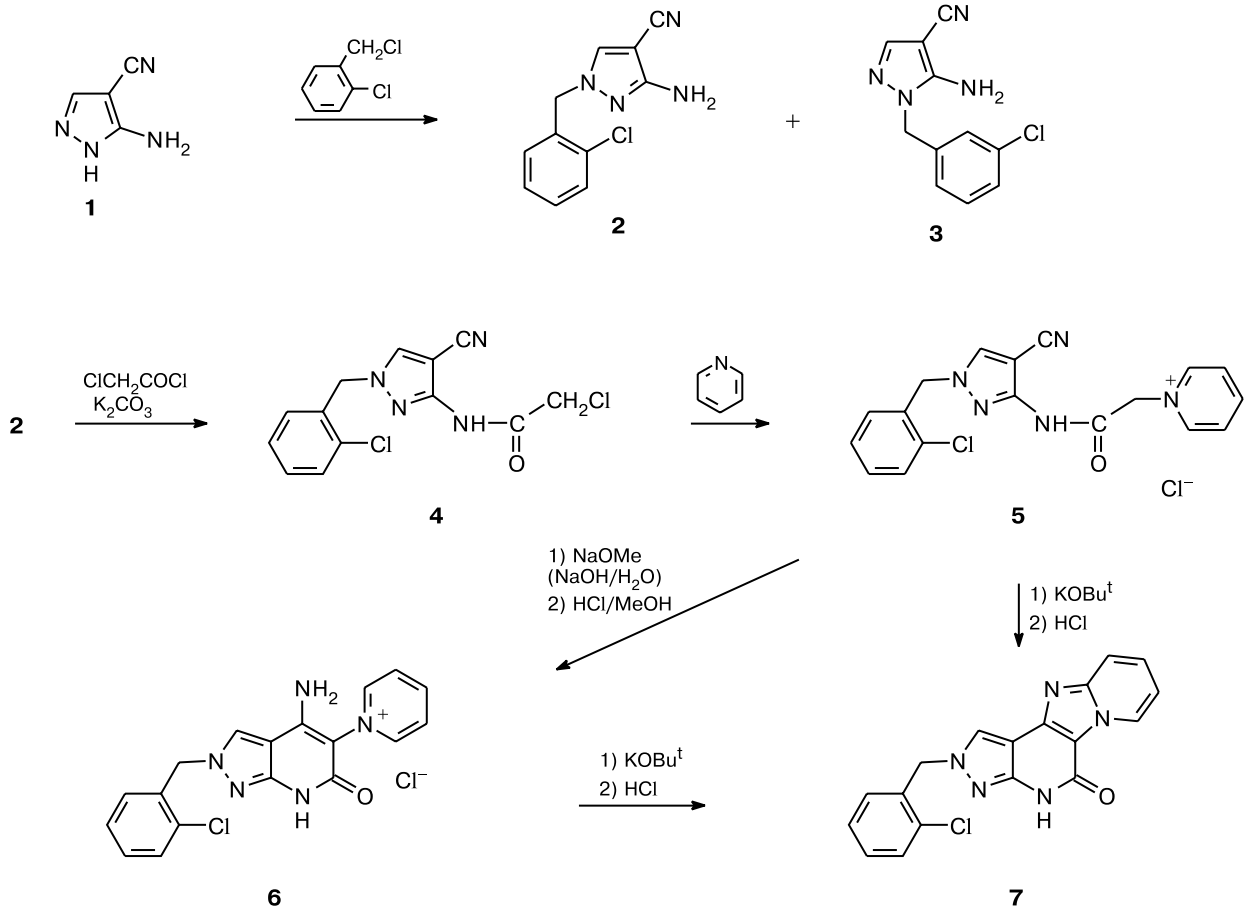
Scheme 1



Chloroacetylation of compound **2** in dioxane in the presence of potassium carbonate afforded the corresponding chloroacetyl derivative **4**, which was used in the further synthesis of the target 4-aminopyrazolo[3,4-*b*]pyridine. Taking into account the possibility of the use of various pyridinium salts[7,8] in heterocyclic synthesis, compound **4** was used in the reaction with pyridine at room temperature to prepare 1-[1-(2-chlorobenzyl)-4-cyanopyrazol-3-ylaminocarbonylmethyl]pyridinium chloride (**5**) (95% yield). A high CH-acidity of the methylene unit located between the electron-withdrawing carbonyl and pyridinium substituents ensures cyclization involving the nitrile group. Actually, the reaction of sodium methoxide with salt **5** in methanol or heating of this salt in aqueous alkaline solution produced pyrazolo[3,4-*b*]pyridinone **6** in 54% yield. The structure of the latter was established by elemental analysis, ^{1}H NMR spectroscopy, and mass spectrometry.

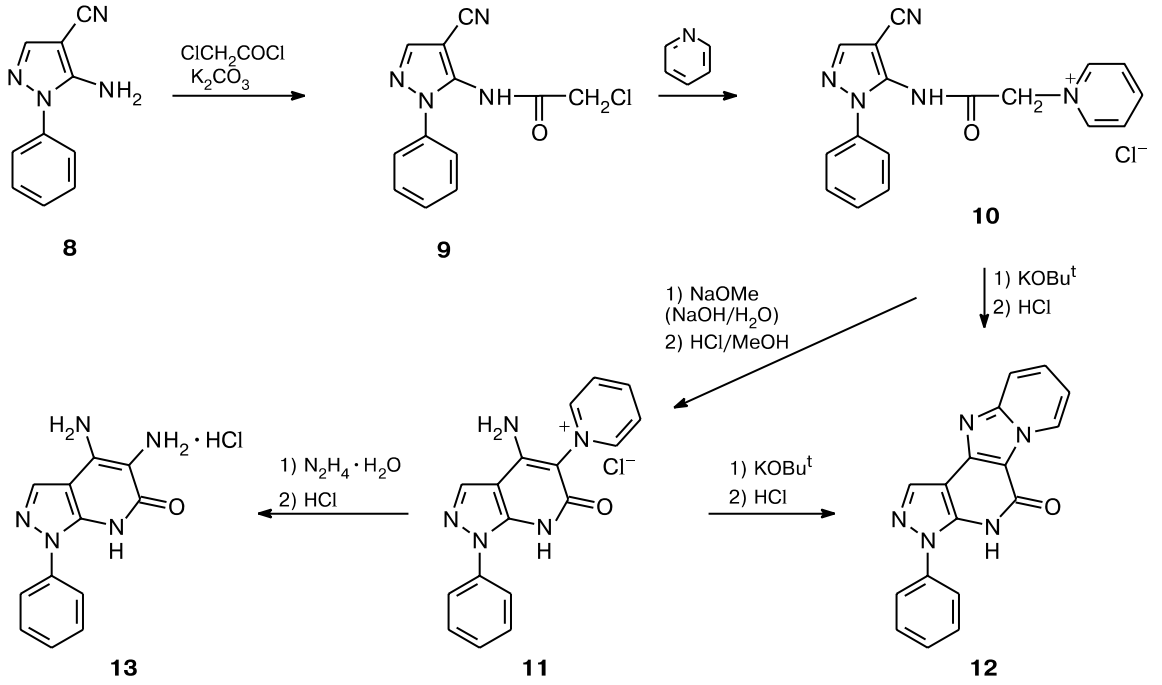
It should be noted that the reaction mixture contained, along with pyridinium chloride **6**, the starting pyrazole **2**, *i.e.*, under these conditions, the reaction at the pyridine nitrogen atom was accompanied by deacylation. In addition, a small amount of yet another compound was present in the reaction mixture. Based on the physicochemical data, we assigned the structure of 2-(2-chlorobenzyl)-2,4-dihydro-5*H*-pyrazolo[3,4-*b*]pyrido[1′,2′:1,2]imidazo[4,5-*d*]pyridin-5-one (**7**) to the latter compound. A further investigation demonstrated that refluxing of pyridinium salt **5** with 2 equiv. of potassium *tert*-butoxide in *tert*-butanol for 2 days afforded tetracyclic compound **7** in 80.5% yield.

Somewhat better results were obtained by refluxing bicyclic pyridinium salt **6** with 2 moles of potassium *tert*-butoxide in *tert*-butanol for 1 day. In this case, compound **7** was synthesized in 90% yield.

In the next step of our investigation, we used 5-amino-4-cyano-1-phenylpyrazole (**8**)[6] as the starting compound analogously to pyrazole **2**. The reaction of compound **8** with chloroacetyl chloride smoothly produced chloroacetyl derivative **9**, whose treatment with pyridine gave 1-(4-cyano-1-phenylpyrazol-5-ylaminocarbonylmethyl)pyridinium chloride (**10**) in 97% yield (Scheme 2).

The reaction of sodium methoxide with salt **10** in methanol or storage of salt **10** in an aqueous alkaline

Scheme 2



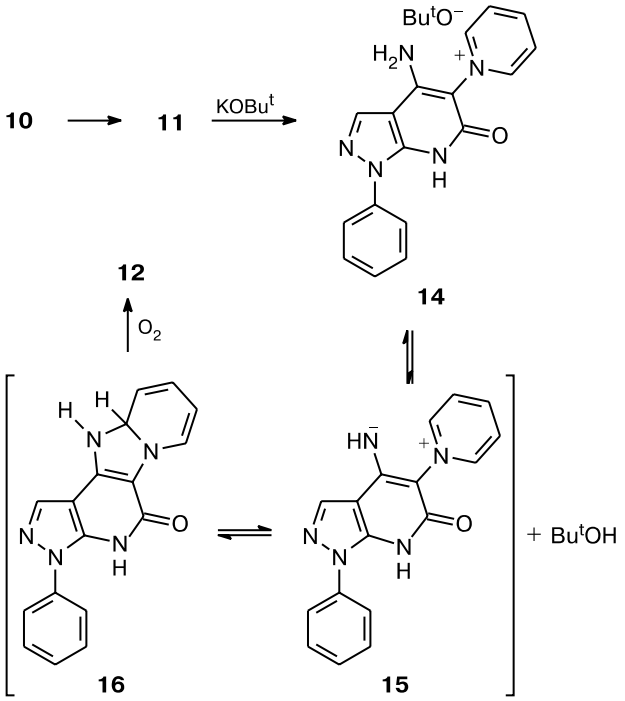
solution afforded 1-[4-amino-6(7*H*)-oxo-1-phenyl-1*H*-pyrazolo[3,4-*b*]pyridin-5-yl]pyridinium chloride (**11**) in 61% yield. Simultaneously, the reaction gave the starting 5-amino-4-cyano-1-phenylpyrazole. Recall that cyclization of salt **5** under the same conditions was also accompanied by analogous deacylation.

Refluxing of pyridinium chloride **10** in *tert*-butanol with a twofold excess of *tert*-potassium butoxide for 8 days afforded 3-phenyl-3,4-dihydro-5*H*-pyrazolo[3,4-*b*]pyrido[1′,2′:1,2]imidazo[4,5-*d*]pyridin-5-one (**12**) in 72% yield. Compound **12** of higher quality was synthesized more rapidly by refluxing pyridinium chloride **11** in *tert*-butanol with 2 moles of potassium *tert*-butoxide. The structures of the reaction products were established by spectroscopic methods.

As can be seen from Schemes 1 and 2, the reactions of both 1-(*o*-chlorobenzyl)-substituted 3-amino-4-cyanopyrazole **2** and 1-phenyl-substituted 5-amino-4-cyanopyrazole **8** follow the same pathway, *i.e.*, these reactions have a general character. We observed the difference in the behavior of intermediates in studies of the transformation of the pyridinium fragment into the amino group typical of pyridinium salts.[6,7,9] We failed to find conditions for this transformation of compound **6**, whereas pyridinium salt **11** was decomposed by heating with 50% aqueous hydrazine hydrate for several days. The subsequent treatment of the product with a methanolic solution of hydrogen chloride afforded 4,5-diamino-6(7*H*)-oxo-1-phenyl-1*H*-pyrazolo[3,4-*b*]pyridine **13** in high yield.

It is worthwhile to discuss the pathway of unusual cyclization giving rise to tetracyclic compounds **7** and **12**. Let us consider the transformation of pyridinium salt **11** as an example (Scheme 3).

Scheme 3



It is reasonable to assume that the synthesis of tetracyclic compound **12** occurs through bicyclic pyridinium salt **11** as the key intermediate (this is also true for the reaction in which monocyclic salt **10** is used as the primary starting compound, which is initially transformed into **11**). The subsequent treatment of pyridinium chloride **11** with potassium *tert*-butoxide afforded compound **14** followed by the proton abstraction from the primary amino group at position 4 of the bicyclic compound giving rise to zwitterion **15**. Apparently, this is the rate-determining step of the process as a whole, which is responsible for the long time of transformation **11** → **12**. The addition of the resulting amide anion at the α position of the pyridine ring afforded dihydro derivative **16**, whose oxidation with atmospheric oxygen gave tetracyclic derivative **12** (tetracyclic compound **7** is generated analogously). It should be noted that the reaction of potassium *tert*-butoxide with pyridinium chloride **10** under argon did not produce tetracyclic compound **12**, *i.e.*, oxidation is a necessary step. If intermediate **16** cannot be oxidized (under anaerobic conditions, when argon is passed through the solution), deacylation of compound **10** giving rise to compound **8** becomes the major reaction.

Experimental

The mass spectra (EI) were obtained on a Finnigan SSQ-710 mass spectrometer using a direct inlet system. The NMR spectra were recorded on a Bruker AC-300 spectrometer in DMSO-d_6. The purity of the reaction products was checked and the course of the reactions was monitored by TLC on Merck TLC Silicagel 60 F_{254} plates (hexane—acetone (3 : 1) as the eluent; chloroform—methanol (10 : 1) with the addition of a drop of ammonia, visualization with UV light). The melting points were determined on an Electrothermal 9100 instrument (UK).

The physicochemical characteristics and elemental analysis data for the reaction products are given in Table 1.

3-Amino-1-(2-chlorobenzyl)-4-cyanopyrazole (2). A suspension of pyrazole **1** (14 g, 13 mmol), *o*-chlorobenzyl chloride (22.8 g, 14.3 mmol), and potassium carbonate (26.8 g, 19.4 mmol) in ethanol (50 mL) was refluxed with stirring for 3 days. The reaction mixture was cooled and then water (500 mL) was added. After 1 day, the yellow precipitate of chlorobenzylpyrazole **2** that formed was filtered off, washed with water, and recrystallized from ethanol.

3-Chloroacetamido-1-(2-chlorobenzyl)-4-cyanopyrazole (4). A solution of chloroacetyl chloride (11.7 g, 10.3 mmol) in dioxane (15 mL) was slowly added dropwise with vigorous stirring to a mixture of chlorobenzylpyrazole **2** (16 g, 6.9 mmol) and potassium carbonate (14.3 g, 10.3 mmol) at 60 °C for 1 h. The reaction mixture was refluxed with stirring for 3 days and cooled. Then water (1000 mL) was added, and the precipitate of **4** that formed was filtered off and recrystallized from ethanol. ^{1}H NMR, δ: 4.26 (s, 2 H, CH_2Cl); 5.42 (s, 2 H, N(1)CH_2); 7.24—7.53 (m, 4 H, C_6H_4); 8.66 (s, 1 H, H(5)); 10.95 (br.s, 1 H, NH).

1-[1-(2-Chlorobenzyl)-4-cyanopyrazol-3-ylaminocarbonylmethyl]pyridinium chloride monohydrate (5). A solution of compound **4** (17 g, 5.5 mmol) in pyridine (170 mL) was stirred at ~20 °C for 3 h, during which a white precipitate was formed in the flask. Isopropyl alcohol (50 mL) was added to the reaction

Table 1. Physicochemical characteristics and elemental analysis data for compounds **2**, **4**—**7**, and **9**—**13**

Compound	Yield (%)	M.p. /°C	Found / Calculated (%)			Molecular formula	MS ([M]$^+$), m/z (I_{rel} (%))
			C	H	N		
2	90	144—146	56.74 56.78	4.03 3.90	23.97 24.08	$C_{11}H_9ClN_4$	232 (47)
4	86	179—180	50.38 50.51	3.31 3.26	17.93 18.12	$C_{13}H_{10}Cl_2N_4O$	309 (9)
5	94	229—231	53.28 53.21	4.24 4.22	17.23 17.24	$C_{18}H_{15}Cl_2N_5O\cdot H_2O$	352 (86)
6	54	355—356	55.84 55.68	4.02 3.89	18.16 18.04	$C_{18}H_{15}Cl_2N_5O$	352 (72)
7	80	358—360	61.82 61.81	3.56 3.46	19.73 20.02	$C_{18}H_{12}ClN_5O$	349 (90)
9	57	149—151	55.28 55.29	3.47 3.48	21.66 21.49	$C_{12}H_9ClN_4O$	260 (33)
10	97	210—212	60.05 60.09	4.19 4.15	20.62 20.61	$C_{17}H_{14}ClN_5O$	304 (14)
11	61	355—357	60.11 60.09	4.19 4.15	19.97 20.61	$C_{17}H_{14}ClN_5O$	304 (87)
12	74	335—337	67.56 67.77	3.76 3.68	23.25 23.24	$C_{17}H_{11}N_5O$	301 (90)
13	70	262—264	51.97 51.90	4.38 4.36	25.29 25.22	$C_{12}H_{12}ClN_5O$	241 (100)

mixture and the mixture was stored in a refrigerator at 5 °C. After 1 day, the precipitate of pyridinium chloride **5** was filtered off, washed with cold isopropyl alcohol, and recrystallized from the same solvent. ^{1}H NMR, δ: 5.44 (s, 2 H, N(1)CH_2); 5.72 (s, 2 H, N(1′)CH_2); 7.29—7.53 (m, 4 H, C_6H_4); 8.21 (m, 2 H, H(3′), H(5′)); 8.65 (s, 1 H, H(5)); 8.69 (m, 1 H, H(4′)); 9.05 (m, 1 H, H(2′), H(6′)); 11.44 (br.s, 1 H, NH).

1-[4-Amino-2-(2-chlorobenzyl)-6(7*H*)-oxo-2*H*-pyrazolo[3,4-*b*]pyridin-5-yl]pyridinium chloride (6). Sodium methoxide (2.8 g, 5.14 mol) was added to a suspension of pyridinium chloride **5** (10 g, 2.57 mmol) in methanol (100 mL). The reaction mixture was heated with stirring for 3 days and cooled. Then a HCl(9%)/MeOH solution and activated carbon were added. The precipitate containing the carbon was filtered off through a funnel with silica gel. The red-brown filtrate was concentrated *in vacuo* to dryness. The precipitate that formed was treated with acetone and filtered off. Pyridinium chloride **6** was isolated by crystallization from isopropyl alcohol. ^{1}H NMR, δ: 5.53 (s, 2 H, N(2)CH_2); 7.27—7.54 (m, 6 H, NH_2, C_6H_4); 8.24 (m, 2 H, H(3′), H(5′)); 8.41 (s, 1 H, H(3)); 8.70 (m, 1 H, H(4′)); 8.96 (m, 2 H, H(2′), H(6′)); 11.54 (br.s, 1 H, NH).

2-(2-Chlorobenzyl)-2,4-dihydro-5*H*-pyrazolo[3,4-*b*]pyrido[1′,2′:1,2]imidazo[4,5-*d*]pyridin-5-one (7). ***A.*** A suspension of pyridinium chloride **6** (5 g, 1.29 mmol) and potassium *tert*-butoxide (2.88 g, 2.58 mmol) in *tert*-butanol (30 mL) was heated with stirring for 1 day. The reaction mixture was cooled and water (100 mL) was added. Then the reaction mixture was acidified with aqueous hydrochloric acid to pH ~5 and stored at 5 °C. After 1 day, the precipitate of **7** that formed was filtered off, washed with water, and recrystallized from DMF.

B. A suspension of pyridinium chloride **5** (2 g, 0.51 mmol) and potassium *tert*-butoxide (1.15 g, 1.03 mmol) in *tert*-butanol was refluxed with stirring for 2 days. The reaction mixture was cooled, water (200 mL) was added, and the mixture was acidified with aqueous hydrochloride acid to pH ~5. The pale-yellow precipitate of **7** that formed was filtered off, washed with water, and twice recrystallized from DMF. ^{1}H NMR, δ: 5.57 (s, 2 H, N(2)CH_2); 7.13, 7.35, and 7.50 (all m, 1 H, 2 H, 1 H, C_6H_4); 7.22 (m, 1 H, H(8)); 7.65 (m, 1 H, H(9)); 7.80 (m, 1 H, H(10)); 8.57 (s, 1 H, H(1)); 9.36 (m, 1 H, H(7)); 11.86 (br.s, 1 H, NH).

5-(2-Chloroacetamido)-4-cyano-1-phenylpyrazole (9). A solution of chloroacetyl chloride (29.2 g, 25.8 mmol) in dioxane (35 mL) was slowly added dropwise with vigorous stirring to a suspension of phenylpyrazole **8** (31.7 g, 17 mmol) in potassium carbonate (35.6 g, 25.8 mmol) at 60 °C for 1 h. The reaction mixture was refluxed with stirring for 3 days and cooled. Then water (1000 mL) was added and $NaHCO_3$ was added to pH ~7. The white precipitate of **9** that formed was filtered off and recrystallized from water. ^{1}H NMR, δ: 4.33 (s, 2 H, CH_2Cl); 7.54 (m, 5 H, Ph); 8.32 (s, 1 H, H(3)); 10.96 (br.s, 1 H, NH).

1-[4-Cyano-1-phenylpyrazol-5-ylaminocarbonylmethyl]pyridinium chloride (10). A solution of compound **9** (5 g, 1.29 mmol) in pyridine (60 mL) was stirred at 40 °C. After 30 min, the white precipitate was obtained. The reaction mixture was continued to be stirred at ~20 °C for 4 h. Pyrazolopyridinium chloride **10** that formed was filtered off, washed with cold isopropyl alcohol, and recrystallized from the same solvent. ^{1}H NMR, δ: 5.89 (s, 2 H, N(1′)CH_2); 7.47—7.71 (m, 5 H, Ph); 8.20 (m, 3 H, H(3′), H(5′), H(3)); 8.68 (m, 1 H, H(4′)); 9.08 (m, 1 H, H(2′), H(6′)); 12.14 (br.s, 1 H, NH).

1-[4-Amino-6(7*H*)-oxo-1-phenyl-1*H*-pyrazolo[3,4-*b*]pyridin-5-yl]pyridinium chloride (11). A suspension of pyridinium chloride **10** (6.1 g, 1.8 mmol) and sodium methoxide (1.94 g, 3.6 mmol) in methanol (100 mL) was refluxed for 3 days. The reaction mixture was cooled and filtered. The filtrate was acidified with a HCl(9%)/MeOH solution to pH ~6, and the inorganic precipitate that formed was filtered off. The resulting filtrate was concentrated *in vacuo* to 20 mL. The precipitate of pyridinium salt **11** was filtered off, washed with methanol, and recrystallized from methanol. ^{1}H NMR, δ: 7.40 (m, 1 H, H(4″); 7.54 (m, 2 H, H(3″), H(5″)); 7.61 (br.s, 2 H, NH_2); 7.83 (m, 2 H, H(2″), H(6″)); 8.30 (m, 2 H, H(3′), H(5′)); 8.46 (s, 1 H, H(3)); 8.72 (m, 1 H, H(4′)); 9.01 (m, 2 H, H(2′), H(6′)); 11.31 (br.s, 1 H, NH(C(5)).

3-Phenyl-3,4-dihydro-5*H*-pyrazolo[3,4-*b*]pyrido[1′,2′:1,2]imidazo[4,5-*d*]pyridin-5-one (12). ***A.*** A suspension of pyridinium salt **11** (5 g, 1.47 mmol) and potassium *tert*-butoxide (3.29 g, 2.94 mmol) in *tert*-butanol (50 mL) was refluxed with stirring for 5 days. The reaction mixture was cooled, water (500 mL) was added, and the mixture was acidified with hydrochloric acid to pH ~5. After 2 h, the precipitate of pyrazolopyridine **12** that formed was recrystallized from DMF.

B. A suspension of pyridinium chloride **10** (5 g, 1.47 mmol) and potassium *tert*-butoxide (4.12 g, 3.68 mmol) in *tert*-butanol (30 mL) was refluxed with stirring for 8 days. The reaction mixture was cooled, water (500 mL) was added, and the mixture was acidified with hydrochloric acid to pH ~5. After 1 day, the precipitate that formed was filtered off and recrystallized from DMF in the presence of carbon; R_f 0.8 (chloroform—methanol, 10 : 1).

Attempts to perform cyclization of salt 10 in the absence of atmospheric oxygen. A suspension of pyridinium chloride **10** (0.3 g, 0.088 mmol) and potassium *tert*-butoxide (0.19 g, 0.17 mmol) in *tert*-butanol (10 mL) was refluxed with stirring under argon for 1 day, after which the TLC test of the reaction mixture showed the presence of pyridinium salt **10** and one unidentified product. Then water (10 mL) was added and the reaction mixture was acidified with hydrochloric acid to pH ~5. The precipitate that formed was filtered off, washed with water, and recrystallized from ethanol in the presence of carbon. 5-Amino-4-cyano-1-phenylpyrazole was obtained in a yield of 0.18 g. ^{1}H NMR, δ: 7.27 (m, 1 H, H(8)); 7.49 (m, 1 H, H(4′); 7.58 (m, 2 H, H(3′), H(5′)); 7.72 (m, 3 H, H(2′), H(6′), H(9)); 7.84 (m, 1 H, H(10)); 8.32 (s, 1 H, H(1)); 9.34 (m, 1 H, H(7)); 12.40 (br.s, 1 H, NH).

4,5-Diamino-6(7*H*)-oxo-1-phenyl-1*H*-pyrazolo[3,4-*b*]pyridine (13). A solution of pyridinium salt **11** (1.4 g, 4.1 mmol) in a 50% aqueous hydrazine hydrate solution was refluxed with stirring for 24 h. The reaction mixture was cooled, diluted with water (140 mL), and carefully acidified with dilute aqueous hydrochloric acid to pH ~7.5. The white precipitate that formed was rapidly filtered off, transferred to a flask, and immediately treated with HCl(9%)/MeOH. Pyrazolopyridine hydrochloride **13** that formed was filtered off and recrystallized from ethanol. ^{1}H NMR, δ: 6.35 (br.s, 2 H, NH_2); 7.27 (m, 1 H, H(4′)); 7.48 (m, 2 H, H(3′), H(5′)); 8.09 (m, 2 H, H(2′), H(6′)); 8.14 (s, 1 H, H(3)).

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