ISSN 1070-4280, Russian Journal of Organic Chemistry, 2008, Vol. 44, No. 2, pp. 257–262. © Pleiades Publishing, Ltd., 2008.
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Synthesis and Properties of Cyanomethyl Derivatives of Imidazo[1,2-*a*]pyridine, Imidazo[1,2-*a*]pyrimidine, and Imidazo[2,1-*b*]thiazole

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Received May 15, 2007

Abstract—2-Cyanomethyl derivatives were obtained of imidazo[1,2-*a*]pyridine, imidazo[1,2-*a*]pyrimidine, and imidazo[2,1-*b*]thiazole, and their reactivity was investigated by an example of imidazo[1,2-*a*]pyridine: It was subjected to nitration, bromination, azo coupling and nitrosation. Acylation of the methylene group effected by amino acids esters with a subsequent addition of the amino group to the cyano group resulted in the formation of 5-amino-4-imidazo[1,2-*a*]-pyridin-2-yl-1-phenyl-1,2-dihydro-3*H*-pyrrol-3-one and 2-amino-1-ethyl-3-imidazo[1,2-*a*]pyridin-2-yl-4(1*H*)-quinolinone.

DOI: 10.1134/S1070428008020127

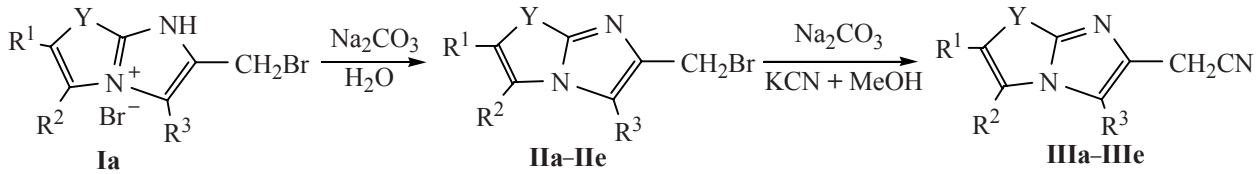
Derivatives of imidazoheterocycles can exhibit a versatile biological activity: Nonsteroid antiphlogistic drugs [1], cardiotonic and antiarrhithmic pharmaceuticals [2], substances with a neurotropic action [3, 4], and those possessing cytoprotective and antitumor activity [5] are found among these compounds. Therefore the development of new preparation procedures for these fused systems is very promising.

The bromomethyl derivatives of imidazo[1,2-*a*]-pyridine, imidazo[1,2-*a*]pyrimidine, and imidazo[2,1-*b*]thiazole **I, II** [6, 7] that we have previously synthesized can be by a known procedure converted into the corresponding cyanomethyl heterocycles **III** [8].

Methylene group in these compounds is less reactive than in the 1-methyl-2-cyanomethylbenzimidazole [9]. It is not acylated with anhydrides and chlorides of carboxylic acids. However in reaction of diazonium salts with compound **IIIa** formed bisazo dyes of **IVa** and **IVb** type (Scheme 2). In the ^{1}H NMR spectra of compounds **IVa** and **IVb** the signal of proton H^5 is shifted downfield by 0.9 ppm. This abnormal paramagnetic shift indicates that these compounds are present prevailingly in the *syn*-conformation [10]. No signals from the position *3* of the bicycle and CH_2 group and also the chemical shifte and intensities of the other signals in the ^{1}H spectrum confirm the assumed structures of the azo dyes **IVa** and **IVb**. At equivalent ratio of the reactant an intractable mixture was obtained containing along with other compounds (TLC data) bisazo dye **IV** and free base **IIIa**.

The nitrosation of imidazo[1,2-*a*]pyridine-2-ylacetonitrile (**IIIa**) led to the formation of 2-(hydroxyimino)(3-nitrosoimidazo[1,2-*a*]pyridine-2-yl)-acetonitrile (**VII**).

Scheme 1.



Ia **IIa–IIe** **IIIa–IIIe**

Ia–IIIa, $R^1 = R^2 = H$, Y = CH=CH, $R^3 = H$; **II, III**, $R^1 = Cl$, $R^2 = H$, Y = CH=CH, $R^3 = Cl$ (**b**); $R^1 = R^2 = H$, Y = CH=CH, $R^3 = Br$ (**c**); $R^1 = H$, $R^2 = CH_3$, Y = CH_3–CN=CH, $R^3 = H$ (**d**); $R^1 = H$, $R^2 = H$, Y = S, $R^3 = Br$ (**e**).

† Deceased.

In the ^{1}H NMR spectrum of this compound a slightly broadened singlet of the hydroxy group proton appeared in the region 15 ppm, and it disappeared at treatment with deuterium oxide. The signal of H^5 proton is shifted downfield by 1.4 ppm compared to the spectrum of the initial compound **IIIa** indicating that this substance is present prevailingly in the *syn*-conformation [10].

The bromination and nitration of compound **IIIa** occurred only in the position *3*. Salt **V** isolated after bromination in dioxane (Scheme 2) treated with 2 M alkali solution was readily converted into (3-bromoimidazo[1,2-*a*]pyridine-2-yl)acetonitrile (**IIIc**).

The nitration of nitrile **IIIa** gave (3-nitroimidazo-[1,2-*a*]pyridine-2-yl)acetonitrile (**VI**).

Neither salt **V** nor base **IIIc** entered into the reactions of azo coupling, nitrosation, bromination, and nitration.

Imidazo[1,2-*a*]pyridine-2-ylacetonitrile (**IIIa**) was acylated at the methylene group with methyl 2-(ethylamino)benzoate in the presence of sodium *tert*-butylate (structure **VIII**), but we failed to isolate this substance for the ethylamino group underwent an intramolecular addition to the cyano group (Scheme 3). The product of the reaction was 2-amino-3-(imidazo[1,2-*a*]pyridine-2-yl)-1-ethyl-4(1*H*)-quinolinone (**X**). Similarly compound **IIIa** reacted with ethyl N-phenylglycinate yielding 5-amino-4-imidazo[1,2-*a*]pyridine-2-yl-1-phenyl-1,2-dihydro-3*H*-pyrrol-3-one (**XIII**).

At the treatment of crude products **X** and **XIII** with dilute hydrochloric acid bishydrochlorides **IX** and **XII** were obtained that on adding aqueous ammonia provided analytically pure 2-amino-(3-imidazo[1,2-*a*]pyridin-2-yl)-1-ethyl-4(1*H*)-quinolinone (**X**) and 5-amino-(4-imidazo[1,2-*a*]pyridine-2-yl)-1-phenyl-1,2-dihydro-3*H*-pyrrol-3-one (**XIII**).

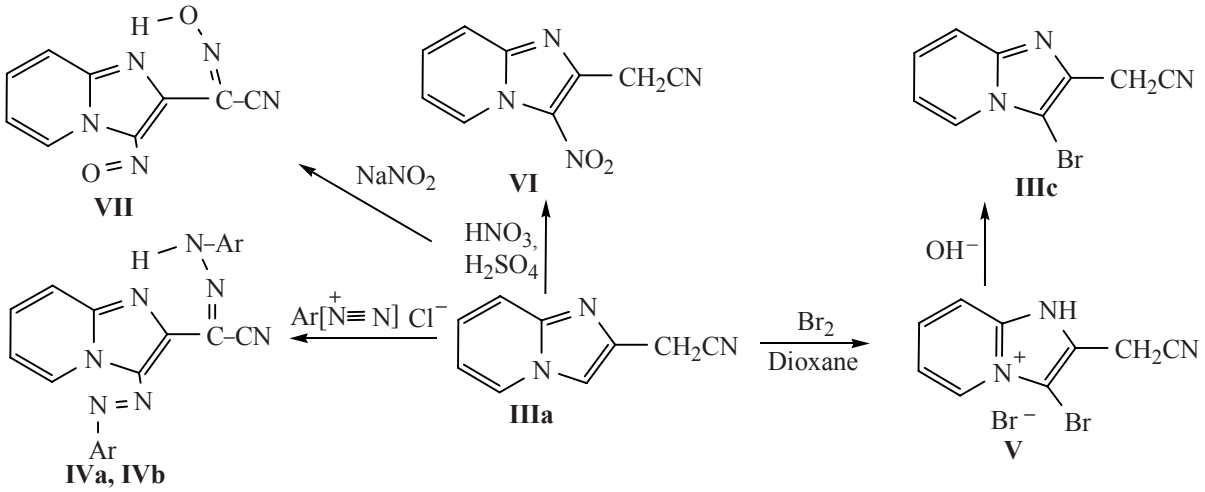
The treatment of 2-amino-3-aryl-4-quinolones in acetic acid solution with sodium nitrite is known to lead to the formation of the derivatives of a quino[2,3-*c*]-cinnolone heterocyclic system [11]. However the treatment of amine **X** with sodium nitrite in dilute hydrochloric acid resulted in the nitrosation in the position *3* of imidazopyridine giving a dihydrochloride of nitroso derivative **XIV** converted into compound **XV** with aqueous ammonia. This reaction route is due to the electron-donor effect of the nitrogen atom in the position *2* of the imidazo[1,2-*a*]pyridine fragment [12]. The intramolecular coordination of the amino group proton in 2-amino-3-(3-nitrosoimidazo[1,2-*a*]pyridine-2-yl)-1-ethyl-4(1*H*)-quinolinone (**XV**) hampers the reciprocal rotation of both heterocycles.

EXPERIMENTAL

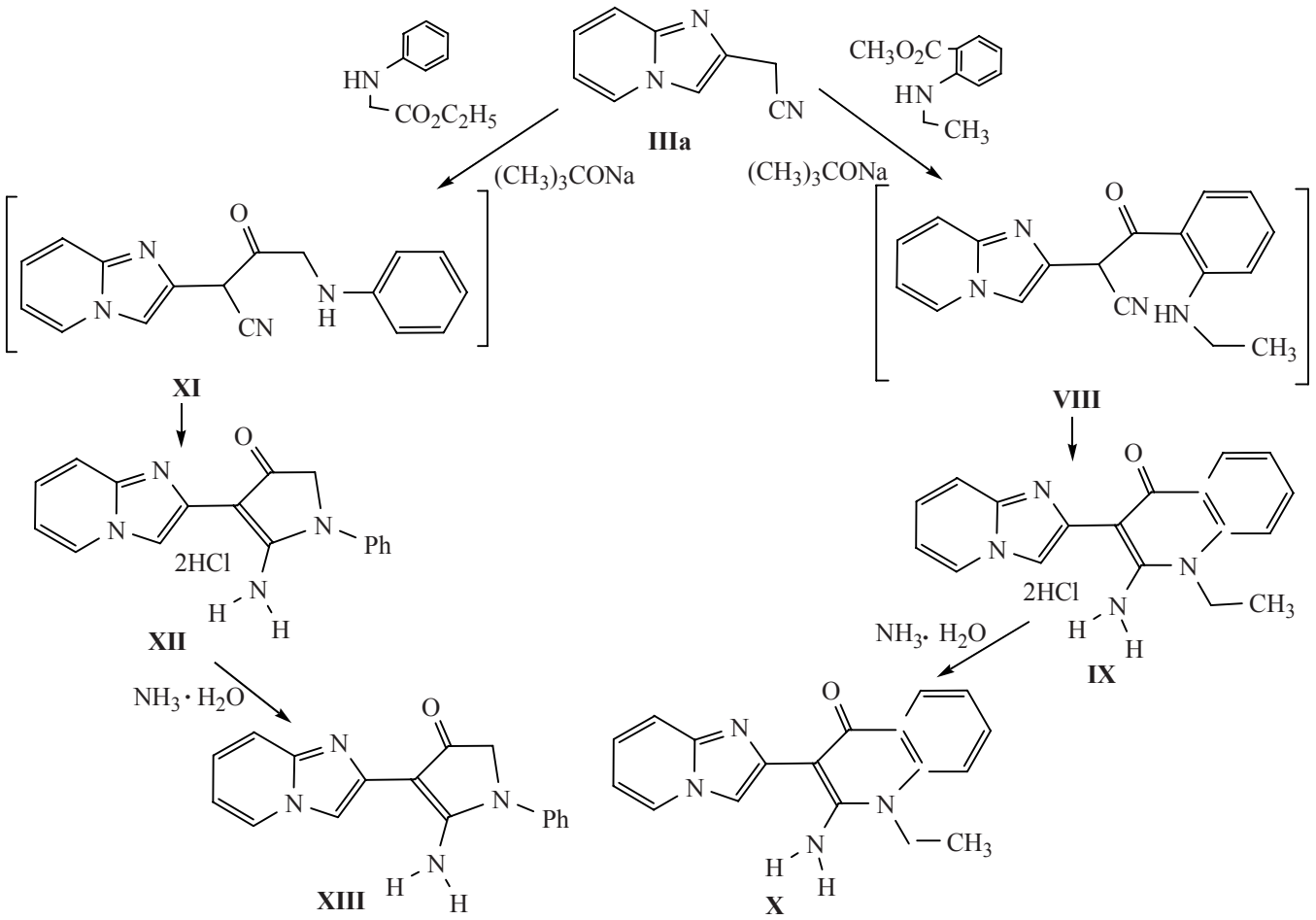
IR spectra were recorded on a spectrophotometer Pye-Unicam SP3-300 from KBr pellets. ^{1}H NMR spectra were registered on a spectrometer Varian Mercury 400 (400 MHz), internal reference TMS. The reaction progress was monitored and the purity of products obtained was checked by TLC on Silufol UV-254 plates.

Imidazo[1,2-*a*]pyridin-2-ylacetonitrile (IIIa). To a dispersion of 21.2 g (0.073 mol) of 2-bromomethyl-1*H*-imidazo[1,2-*a*]pyridinium bromide in 140 ml of methanol a solution was added of 7.42 g (0.07 mol) of sodium hydrogen carbonate in 56 ml of water. A colorless precipitate formed, the mixture was stirred for 5 min, then 7.6 g (0.12 mol) of potassium cyanide was added.

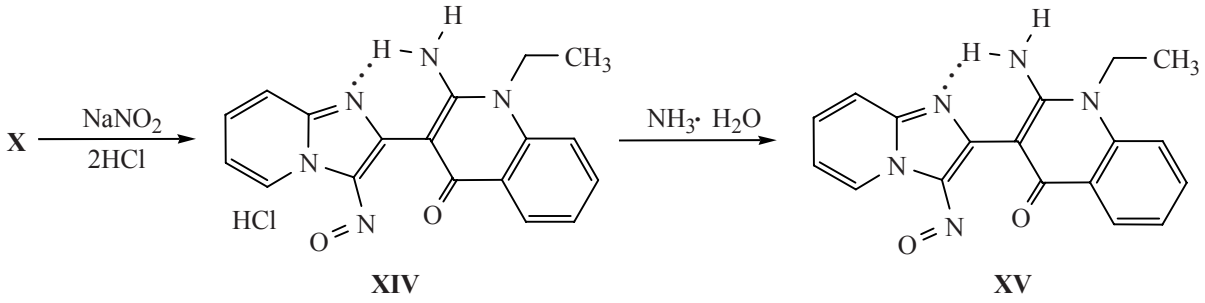
Scheme 2.



Scheme 3.



Scheme 4.



The stirring was continued for 6 h, the precipitate was filtered off and washed with ethanol. The mother liquor was evaporated to dryness, and the reaction product was extracted with ether from the dry residue. The insoluble residue was alkalinized with 2 M solution of NaOH and several times treated with ether. The ether was distilled off on a rotary evaporator, the crystals of the nitrile were dried and recrystallized from 2-propanol. Yield 5.72 g (51%), mp 91–92°C (92°C [8]). IR spectrum, ν, cm^{-1}: 2254 (CN). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 8.47 d (1H, *J* 6.1 Hz), 7.88 s (1H), 7.46 d (1H, *J* 8.8 Hz), 7.22 m (1H), 6.84 m (1H), 3.99 s (2H). Found, %: C 68.56; N 26.54. $C_9H_7N_3$. Calculated, %: C 68.78; N 26.74.

Compounds IIIb–IIIe. *General procedure.* To 0.1 mol of base **II** in 140 ml of methanol 7.6 g (0.12 mol) of

potassium cyanide was added. The mixture was stirred for 5–6 h, the precipitate was filtered off and washed with ethanol. The mother liquor was evaporated to dryness, and the nitrile was extracted with ether from the dry residue. The insoluble residue was alkalinized with 2 M solution of NaOH and several times treated with ether. The ether was distilled off on a rotary evaporator.

(3,6-Dichloroimidazo[1,2-*a*]pyridin-2-yl)acetonitrile (IIIb). Yield 8.54 g (50%), mp 130°C (ethanol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 8.39 s (1H), 7.66 d (1H, *J* 9.6 Hz), 7.37 d (1H, *J* 9.6 Hz), 4.06 s (2H, CH_2). Found, %: C 47.45; Cl 31.22; N 18.14. $C_9H_5Cl_2N_3$. Calculated, %: C 47.82; Cl 31.37; N 18.59.

(3-Bromoimidazo[1,2-*a*]pyridin-2-yl)acetonitrile (IIIc). *a*. Yield 9.56 g (56%), mp 149°C (2-propanol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 8.25 d (1H, *J* 6.8, *J* 9.6 Hz), 7.59 d (1H, *J* 9.2 Hz), 7.36 m (1H), 7.08 m (1H), 4.03 s (2H, CH_2). Found, %: Br 34.52; N 17.60. $C_9H_6BrN_3$. Calculated, %: Br 33.85; N 17.80.

b. To 0.64 g (0.002 mol) of salt **V** in 10 ml of water was added at room temperature 2 M solution of NaOH till pH 8. The separated precipitate was filtered off, washed with water, dried, and crystallized from 2-propanol. Yield 0.43 g (99%), mp 149°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 8.25 d (1H, *J* 6.8 Hz), 7.59 d (1H, *J* 9.2 Hz), 7.36 m (1H), 7.08 m (1H), 4.03 s (2H, CH_2). Found, %: Br 34.68. N 17.67. $C_9H_6BrN_3$. Calculated, %: Br 33.85; N 17.80.

(5,7-Dimethylimidazo[1,2-*a*]pyrimidin-2-yl)acetonitrile (IIId). Yield 9.63 g (68%), mp 184–186°C (ethanol). IR spectrum, ν, cm^{-1}: 2235.99 (CN). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 7.88 s (1H), 6.84 s (1H), 3.99 s(2H, CH_2), 2.57 s (6H, CH_3 and CH_3). Found, %: C 64.56; N 30.27. $C_{10}H_{10}N_4$. Calculated, %: C 64.50; N 30.09;.

(5-Bromoimidazo[2,1-*b*][1,3]thiazol-6-yl)acetonitrile (IIIe). Yield 7.18 g (46%), mp 110°C (2-propanol). IR spectrum, ν, cm^{-1}: 2245 (CN). ^{1}H NMR spectrum (DMSO–d_6), δ, ppm: 7.72 d (1H, *J* 4.8 Hz), 7.38 d (1H), 3.86 s (2H, CH_2). Found, %: N 17.36; S 13.24. $C_7H_4BrN_3S$. Calculated, %: N 17.47; S 13.63.

Azo dyes IVa and IVb. *General procedure.* The diazonium salt was obtained at 0°C from 7.6 mmol of amine, 0.54 g (7.8 mmol) of sodium nitrite, 1.91 ml (22.8 mmol) of hydrochloric acid, and 6.34 ml of water, sodium acetate was added till neutral reaction toward Congo, and the mixture obtained was added to the ethanol solution of 0.52 g (3.3 mmol) of nitrile (**IIIa**) cooled on an ice bath. After 1 h 9 mg (0.1 mmol) of Na_2CO_3 was added, the separated precipitate was filtered off, washed with ethanol, dried, and crystallized.

(2*Z*)-{3-[(*E*)-Phenyldiazenyl]imidazo[1,2-*a*]-pyridin-2-yl}(phenylhydrazono)acetonitrile (IVa). Yield 1.14 g (72%), mp 191°C (decomp.) (aqueous DMF). IR spectrum, ν, cm^{-1}: 2212.39 (CN). ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 9.30 d (1H, *J* 5.6, *J* 9.6 Hz), 8.82 t (1H, *J* 8.8 Hz), 8.37 d (1H, *J* 8.4 Hz), 8.21 m (2H, H^o, PhN=N), 8.05 t (1H, *J* 6.5 Hz), 7.93–7.71 m (8H, protons PhNH, H^{m,p}, PhN=N). Found, %: C 69.25; N 26.97. $C_{21}H_{15}N_7$. Calculated, %: C 69.03; N 26.83.

2-{3-[(*E*)-2-(4-Methoxyphenyl)diazenyl]-imidazo-[1,2-*a*]pyridin-2-yl}-2-[(*Z*)-2-(4-methoxyphenyl)-hydrazono]acetonitrile (IVb). Yield 1.00 g (65%), mp 288°C (decomp.). IR spectrum, ν, cm^{-1}: 2218 (CN), 1722 (C=O). ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 9.36 d (1H, *J* 5.6 Hz), 8.88 t (1H, *J* 8.8 Hz) 8.65 m (2H, H^o, PhN=N), 8.47–8.34 m (5H, H$^{m,m'}$, PhN=N, H$^{\delta}$), 8.12–8.05 t (3H, H$^{o'}$, PhN=N, *J* 6.5 Hz), 4.27 s (3H, OCH_3), 4.23 s (1H, OCH_3). Found, %: C 62.56; N 20.43. $C_{25}H_{19}N_7O_4$. Calculated, %: C 62.37; N 20.36.

3-Bromo-2-(cyanomethyl)-1*H*-imidazo[1.2-*a*]-pyridin-4-ium bromide (V). To 0.8 g (5 mmol) imidazo[1,2-*a*]pyridin-2-ylacetonitrile (**IIIa**) in 20 ml of dioxane was added at room temperature 0.26 ml (5 mmol) of bromine, and the mixture was stirred for 4 h. The separated precipitate was filtered off, washed with dioxane, dried, and crystallized from 2-propanol. Yield 0.98 g (93%), mp 142°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 8.40 d (1H, *J*6.8 Hz), 7.74 d (1H, *J* 8.8 Hz), 7.56 m (1H), 7.25 m (1H), 5.4 br.s (H_2O, exchange with a proton), 4.16 s (2H, CH_2). Found, %: C 34.19; Br 50.52; N 13.31. $C_9H_7Br_2N_3$. Calculated, %: C 34.10; Br 50.42; N 13.26.

(3-Nitroimidazo[1,2-*a*]pyridin-2-yl)acetonitrile (VI). To a nitrating mixture composed of 0.5 ml (7.5 mmol) of 70% nitric acid and 10 ml of concn. H_2SO_4 was added 0.8 g (5 mmol) of nitrile **IIIa**, and the reaction mixture was stirred for 3 h at room temperature. Then it was poured on ice, and 12 mg (0.1 mmol) of sodium sulfate was added. The separated precipitate was filtered off, washed with water, dried, and crystallized. Yield 0.14 g (18%), mp 173°C (decomp.) (2-propanol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 9.4 d (1H, *J* 6.8 Hz), 7.96 d (1H, *J* 9.2 Hz), 7.84 m (1H), 7.50 m (1H), 4.52 s (2H, CH_2). Found, %: C 53.40; N 28.01. $C_9H_6N_4O_2$. Calculated, %: C 53.47; N 27.71.

(2)-(Hydroxyimino)(3-nitrosoimidazo[1,2-*a*]-pyridin-2-yl)acetonitrile (VII). To 0.39 g (0.025 mol) of

nitrile **IIIa** in 4 ml of glacial acetic acid was added by small portions a solution of 0.2 g (0.029 mol) of sodium nitrite in 1 ml of water. The reaction proceeded for 15 min, then 8–10 ml of water was added. The separated precipitate was filtered off, washed with water, and dried. To the mother liquor 0.5 g of additional dry sodium nitrite was added. The separated precipitate was identical to the preceding portion. Yield 0.42 g (79%), mp 236°C (aqueous DMF). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 15.05 br.s (1H, OH), 9.70 d (1H, *J* 6.8 Hz), 8.09 m (2H), 7.58 m (1H). Found, %: C 50.57; N 32.41. $C_9H_5N_5O_2$. Calculated, %: C 50.24; N 32.55.

2-Amino(3-imidazo[1,2-*a*]pyridin-2-yl)-1-ethyl-4(1*H*)-quinolinone dihydrochloride (X). A mixture of 1.57 g (1 mmol) of nitrile **IIIa**, 2.17 g (1 mmol) of ethyl N-ethylanthranilate, and 4 g of sodium *tert*-butylate was boiled in 30 ml of pyridine, the pyridine was distilled off in a vacuum, and the residue was ground in 100 ml of water. To the reaction mixture 27 ml of 3.5% HCl was added at stirring, and some hours later the precipitate obtained was filtered off. Yield 2.02 g (57%), mp 190°C. IR spectrum, ν, cm^{-1}: 1651(C=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 9.08 d (1H, *J* 6.71 Hz*), 8.66 s (1H*), 8.44 br.s (2H, NH_2), 8.24 d (1H, *J* 7.93 Hz*), 7.95 m (4H), 7.45 m (2H), 4.32 t (2H, CH_2, *J* 7.32 Hz), 1.35 t (3H, CH_3). Found, %: N 14.92; Cl 18.97. $C_{18}H_{18}Cl_2N_4O$. Calculated, %: N 14.85; Cl 18.79.

2-Amino-(3-imidazo[1,2-*a*]pyridin-2-yl)-1-ethyl-4(1*H*)-quinolinone (XI). In water was dissolved 0.5 g (0.0013 mol) of salt **X**, a base was added, and some hours later the precipitate obtained was filtered off. Yield 0.29 g (73%), mp 139–140°C. IR spectrum, ν, cm^{-1}: 1626 (C=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 9.36 br.s (2H, NH_2), 9.21 s (1H*), 8.66 d (1H, *J* 6.71 Hz*), 8.32 d (1H, *J* 7.93 Hz*), 7.61 m (2H**), 7.28 m (2H*,**), 6.90 m (1H*), 4.32 t (1H, CH_2-N, *J* 7.2 Hz), 1.35 t (3H, CH_3). Calculated, %: C 71.04; N 18.41. $C_{18}H_{16}N_4O$. Found, %: C 71.21; N 18.68;.

5-Amino-4-imidazo[1,2-*a*]pyridin-2-yl-1-phenyl-1.2-dihydro-3*H*-pyrrol-3-one dihydrochloride XIII). A mixture of 1.57 g (1 mmol) of nitrile **IIIa**, 1.90 g (0.01 mol) ethyl *N*-phenylaminoacetate, and 4 g of sodium *tert*-butylate was boiled in 30 ml of pyridine for 2.5 h. The pyridine was distilled off in a vacuum, and the residue was ground in 100 ml of water. To the reaction mixture 3 ml of acetic acid was added at stirring, and some hours later the precipitate obtained was filtered off, the precipitate was dissolved in dilute hydrochloric acid (1:10), and some hours later the precipitate was filtered off. Yield 1.8 g (52%), mp 242°C. IR spectrum, ν, cm^{-1}: 1623–1658 (C=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 8.80 d (1H, *J* 5.8 Hz*), 8.76 s (1H*), 8.02 d (2H, NH_2), 8.99 d (1H, *J* 8.8 Hz*), 7.74 t (1H*), 7.51 t (2H, H^{m}Ph), 7.45 d (2H, H^{O}Ph, *J* 8.8 Hz), 7.36 m (2H, CH and H^{n}Ph), 4.36 C (2H, CH_2). Found, %: C 56.34; Cl 19.71; N 15.56. $C_{17}H_{16}Cl_2N_4O$. Calculated, %: C 56.21; Cl 19.52; N 15.42.

5-Amino(4-imidazo[1,2-*a*]pyridin-2-yl)-1-phenyl-1,2-dihydro-3*H*-pyrrol-3-one (XIV). A solution of 0.5 g (1.4 mmol) of salt **XIII** in water was neutralized with a water solution of sodium hydrogen carbonate, the precipitate was filtered off. Yield 0.15 g (38%), mp 185°C. IR spectrum, ν, cm^{-1}: 1578 (C=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 8.55 d (1H, *J* 6.4 Hz*), 8.32 s (1H*), 8.01 br.s (2H, NH_2), 7.62 d (1H, *J* 8.8 Hz*), 7.49 t (1H*), 7.45 m (3H, H^{m,n}Ph), 7.30 d (2H, H^{O}Ph, *J* 8.8 Hz), 4.20 s (2H, CH_2). Found, %: C 70.63; N 19.38. $C_{17}H_{14}N_4O$. Calculated, %: C 70.33; N 19.30.

2-Amino-1-ethyl-3-(3-nitrosoimidazo[1,2-*a*]-pyridin-2-yl)-4(1*H*)-quinolinone dihydrochloride (XV). To 1 g (3 mmol) of amine **XI** in dilute hydrochloric acid was added 0.3 g (4.3 mmol) of sodium nitrite. Some hours later the precipitate obtained was filtered off, washed with acetone and ether. Yield 0.77 g (80%), mp 201°C (decomp.) (2-propanol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 9.76 d (1H, *J*6.71*), 8.64 br.s (2H, NH_2), 8.17 d (1H, *J* 7.93 Hz*), 8.14 m (2H**), 7.00 m (2H**), 7.61m (2H*), 4.54 s (2H, CH_2), 1.39 t (3H). Found, %: C 53.32; Cl 17.56; N 17.38. $C_{18}H_{17}Cl_2N_5O_2$. Calculated, %: C 53.21; Cl 17.45; N 17.24.

2-Amino-1-ethyl-3-(3-nitrosoimidazo[1,2-*a*]-pyridin-2-yl)-4(1*H*)-quinolinone (XVI). To a solution of 0.5 g (1.2 mmol) of salt **XV** in water several drops of aqueous ammonia was added, and the precipitate formed was filtered off. Yield 0.24 g (58%), mp 229°C (decomp.). IR spectrum, ν, cm^{-1}: 1614 (C=O). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 9.80 d (1H, *J* 8.00 Hz*), 7.95 m (2H*), 7.63 m (2H**), 7.49 br.s (2H, NH_2), 7.41 m (2H*), 7.29 m (1H**), 4.34 s (2H, CH_2), 1.43 t (3H, CH_3). Found, %: C 64.97; N 21.22. $C_{18}H_{15}N_5O_2$. Calculated, %: C 64.86; N 21.02.

The signals from the imidazo[1.2-*a*]pyridine ring are marked with one asterisk, from the quinolone fragment, with two asterisks.

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