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Features of 2-, 3-Aminopyridines and 2-Aminopyrimidine Condensation with 2-Formylimidazoles

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Abstract—Condensation of 2-, 3-aminopyridines and 2-aminopyrimidine with 1-vinyl- and 1-ethylimidazole-2-carbaldehydes and 1-vinyl-, 1-ethylbenzimidazole-2-carbaldehydes takes two paths depending on the reagents structure: either form Schiff bases or aminals of the corresponding heterocyclic carbaldehydes.

The extensive search for easy and unconventional synthetic procedures for preparation of versatile azole and azine derivatives are obviously caused by their presence as building blocks both in synthetic and endogenic biologically active compounds [1–4]. Besides these classes of heterocycles attract stable interest as polydentate ligands for synthesis of various metal complexes significantly extending the range of the pharmacological activity of compounds from azole and azine series [5–7], and also the variety of potential catalysts. In continuation of the studies on condensation of amines and aldehydes of heterocyclic series [8–10] we report here on results of investigation of reaction between 1-vinyl(ethyl)-2-formylimidazoles (**I–IV**) with 2-, 3-aminopyridines (**V, VI**) and 2-aminopyrimidine (**VII**). The main goal was elucidating the effect of the heterocycle structure and the character of its substituents on the direction of the process because of the nucleophilic addition of primary amines to aromatic aldehydes was known to furnish not only Schiff bases but also aminals of the corresponding aldehydes [11, 12].

As a result of these studies we established that unlike the previously performed condensations of azoles aminoderivatives with aldehydes **I–IV** which yielded only azomethines [8–10], 2-aminoazines **V–VII** addition to imidazolecarbaldehydes can furnish as final products alongside Schiff bases also aminals depending on the structure of initial reagents (Scheme 1). For instance, the reaction of 3-aminopyridine **VI** with imidazolecarbaldehydes **I, II** resulted exclusively in new azomethines, 3-[1-vinyl-(ethyl)imidazol-2-yl]methyleneaminopyridines (**VIII, IX**). The reaction proceeded under fairly severe conditions: at melting (~95°C) of equimolar amounts of original reagents within 8–16 h bases **VIII**, **IX** formed in 60–70% yield. At boiling aldehydes **I, II** with pyridine **VI** in various organic solvents (benzene, ethanol, ether) arose an intractable mixture of reaction products and initial reagents.

Benzimidazolecarbaldehydes **III, IV** react differently with 3-aminopyridine (**VI**). With 2-formyl-1-ethylbenzimidazole (**IV**) formed azomethine **XI** in 62% (in benzene) or 70% yield (at melting).

At the same time the reaction of 1-vinyl-2-formylbenzimidazole (**III**) with amine **VI** terminated at the stage of the intermediate hemiaminal **Xa** that precipitated from the solution (in benzene or ether) in nearly quantitative yield (85–90%). We failed to shift the equilibrium to the expected base **X** by changing the reaction conditions. Only at long heating (26 h) of the initial reagents **III** and **VI** in benzene we detected by ^{1}H NMR spectrum formation of about 20% of azomethine **X** alongside quite a number of unidentified reaction products.

In contrast to 3-aminopyridine (**VI**) reaction of its 2-isomer **V** with aldehydes **I–IV** takes two routes providing azomethines and aminals simultaneously, and the reaction products ratio depends essentially on the azole (**I–IV**) nature (Scheme 2).

At heating in benzene equimolar amounts of 2-aminopyridine (**V**) and 1-vinyl(ethyl)-2-formylimidazoles (**I, II**) mostly azomethines **XII, XIII** were isolated in low yield (~30%). The insignificant amounts of aminals **XVI, XVII** (up to 5%) were detected by ^{1}H NMR spectra. Under similar conditions 2-formylbenzimidazoles **III, IV** with 2-aminopyridine afford prevailingly aminals 2-[1-vinyl(ethyl)-

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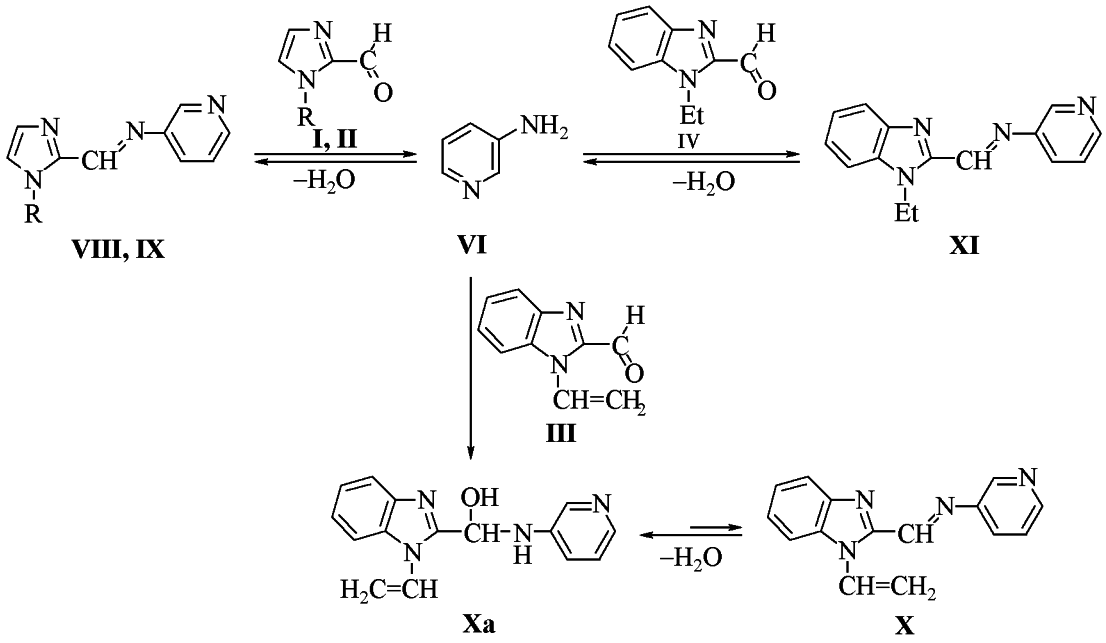
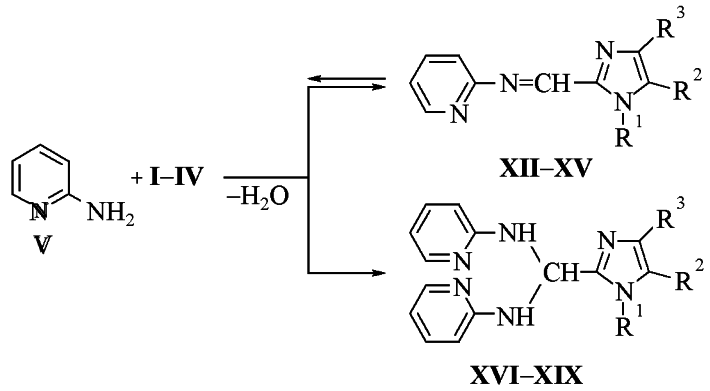
Scheme 1.



I, VIII, R = $CH=CH_2$; **II, IX**, R = Et.

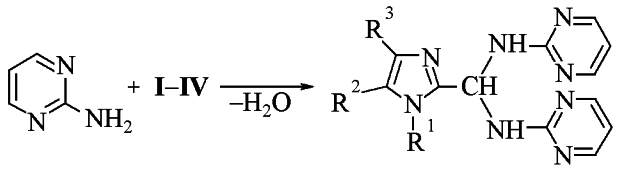
Scheme 2.



XII, XVI, R^1 = $CH=CH_2$, R^2 = R^3 = H; **XIII, XVII**, R^1 = Et, R^2 = R^3 = H; **XIV, XVIII**, R^1 = $CH=CH_2$, R^2, R^3 = $(CH)_4$; **XV, XIX**, R^1 = Et, R^2, R^3 = $(CH)_4$.

benzimidazol-2-yl]methylenediaminopyridines (**XVIII, XIX**) in an yield not exceeding 30–40%, and the corresponding azomethines **XIV, XV** as minor products (3–5%). At the increased reaction time (24 h) and at melting of the initial reagents at ~95°C both reaction paths are conserved but the yield of azomethines **XII, XIII** grows to 45 and 64% respectively, and that of aminals **XVIII, XIX** falls to 10–17%. Therewith in both cases the amount of aminals **XVI, XVII** and azomethines **XIV, XV** arising also as side products remains the same (~5%). At double excess of 2-aminopyridine (**V**) and condensation under conditions of base catalysis (KOH, benzene) we succeeded in obtaining aminals **XVIII, XIX** in 69% yield. As in the general case [11, 12] azomethines formation in the reaction under study is reversible, and in water solutions it strongly shifts to the initial compounds. Aminals apparently arise from addition of the second amine molecule to the intermediately forming hemiaminal, for in reaction of the corresponding azomethine with amine we failed to obtain the expected aminal, and only the initial reagents were recovered.

The condensation of 2-aminopyrimidine (**VII**) with imidazolecarbaldehydes **I–IV** at heating to 60–70°C in ethanol results exclusively in previously unknown aminals 2-[vinyl(ethyl)imidazol-2-yl]methylenediaminopyrimidines (**XX–XXIII**). The maximum yields (57 and 76% respectively) of aminals **XX, XXI** was obtained from 2-formylimidazoles **I, II** in ethanol in 20 h. Under similar conditions the yields of diamines **XXII, XXIII** prepared from benzimidazolecarbaldehydes **III, IV** amount only to 25–36%. The use as solvent of methanol, benzene, dioxane, or condensation in reagents melt, or changing the ratio pyrimidine–aldehyde to 2:1 resulted in decreased yield of compounds **XX–XXIII**.



XX–XXIII

XX, R^1 = $CH=CH_2$, R^2 = R^3 = H; **XXI**, R^1 = Et, R^2 = R^3 = H; **XXII**, R^1 = $CH=CH_2$, R^2, R^3 = $(CH)_4$; **XXIII**, R^1 = Et, R^2, R^3 = $(CH)_4$.

The attempt to prepare aminals **XX–XXIII** under conditions of base catalysis (KOH, K_2CO_3) failed: the isolated mixture according to ^{1}H NMR data contained up to 7% of the corresponding aminal **XX–XXIII** and the initial reagents.

Thus the direction of nucleophilic additions to carbonyl group of imidazolecarbaldehydes **I–IV** of 2-, 3-aminopyridines and 2-aminopyrimidine are essentially governed by azine character. With low-basic 2-aminopyrimidine (**VII**) condensation affords only aminals of heteroaldehydes. 2-Aminopyridine (**V**) reacts with 2-formylimidazoles along two paths giving rise simultaneously to azomethines and aminals in a ratio depending on the electronic structure of the aldehyde component. 3-Aminopyridine (**VI**) with highly basic heterocyclic carbaldehydes **I, II, IV** [13] yields azomethines. The reaction of less basic aldehyde of this series, 1-vinyl-2-formylbenzimidazole (**III**), with amine **VI** is incomplete and is finished at the stage of the corresponding hemiaminal **Xa** formation. It is presumable that like the hydration process of the corresponding aldehydes **I, III** [14], the dehydration at the stage of hemiaminal conversion into azomethine is in some way catalyzed by the heterocyclic fragment of the carbaldehyde and therefore occurs more readily with heterocycles **I, II, IV** as stronger bases.

Azomethines synthesized **VIII, IX, XI** are yellow crystalline compounds, and Schiff bases **X, XII, XIII** are yellow oily substances. Aminals **XVIII–XXIII** are colorless crystals, sparingly soluble in most organic solvents, save compounds **XVIII, XIX**. Hemiaminal **Xa** is a colorless powder insoluble in benzene, ether, and in ethanol, chloroform, carbon tetrachloride, and DMSO it immediately dissociates into the initial amine **VI** and aldehyde **III**. The structure of compounds synthesized was established relaying on ^{1}H NMR, IR, UV, and mass spectra. Characteristics of the compounds are listed in Table 1.

In the IR spectra of azomethines **VIII–XIII** the absorption bands of the stretching vibrations of the azomethine bond ν(N=CH) is observed in the more shortwave region (1630–1615 cm^{-1}) that the corresponding band in the spectra of azomethines prepared from the same aldehydes **I–IV** and aminoazoles (1602–1598 cm^{-1}). Therewith the bands of stretching and bending vibrations of NH_2 group of azines **V, VI** appearing respectively at 3450–3300 and 1640–1630 cm^{-1} are absent in the spectra of bases **VIII–XIII**. Unlike the spectrum of 3-aminopyridine (**VI**) containing a broad band at 3410–3310 cm^{-1} of NH_2 group vibrations, in the IR spectrum of hemiaminal **Xa** a narrow strong band is observed at 3375 cm^{-1} and a wide band with a maximum at 3055 cm^{-1}; the former band corresponds to NH vibrations, the latter to those of associated hydroxy group. The conservation of the vinyl substituent in **Xa** molecule is confirmed by the presence in the IR spectrum of absorption bands at ν 1643 and δ 975 cm^{-1}.

The IR spectra of aminals **XVIII–XXIII** are characterized by a narrow strong absorption band of NH groups in the region 3235–3210 cm^{-1}. The stretching vibrations of the rings belonging to amine and aldehyde moieties appear as very strong absorption bands at 1600 and 1585 cm^{-1} which correspond to overall absorption from aromatic and heterorings. It should also be noted that the absorption bands of vinyl groups in all compounds obtained from 1-vinylimidazole(benzimidazole)carbaldehydes **I, III** conserve their position (ν 1650–1640 and δ 960–950 cm^{-1}).

Difference in UV spectra of azomethines and aminals reflects specific features of their structure. For instance, the spectrum of aminal **XXI** appears as superimposed spectra of the corresponding azole **III** and 2-aminopyrimidine (**VII**). The longwave band in the solution spectrum at 34150 cm^{-1} is observed virtually at the same frequency as the band of

Table 1. Yields and characteristics of compounds synthesized

Compd. no.	Yield, %	mp, °C [bp, °C] (*p*, mm Hg)	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
VIII	62	95–97	66.49	5.04	28.21	$C_{11}H_{10}N_4$	66.67	5.05	28.28
IX	72	64–66	65.83	6.38	27.96	$C_{11}H_{12}N_4$	66.00	6.00	28.00
Xa	83	97–99	68.36	5.25	21.53	$C_{15}H_{13}N_4O$	67.92	4.91	21.13
XI	62	126–128	71.74	5.30	22.42	$C_{15}H_{13}N_4$	72.00	5.60	22.40
XII	45	62–63 [184–186 (3)]	66.33	5.44	28.39	$C_{11}H_{10}N_4$	66.67	5.05	28.28
XIII	64	[186–187 (5)]	65.58	6.09	28.02	$C_{11}H_{12}N_4$	66.00	6.00	28.00
XVIII	69	119–121	69.94	5.50	24.61	$C_{20}H_{18}N_6$	70.18	5.26	24.56
XIX	61	151–153	69.42	6.49	24.27	$C_{20}H_{20}N_6$	69.77	5.80	24.42
XX	57	219–221	57.14	4.76	38.10	$C_{14}H_{14}N_8$	57.35	4.73	38.36
XXI	76	182–185	56.35	5.73	37.36	$C_{14}H_{16}N_8$	56.76	5.41	37.84
XXII	25	193–195	62.57	4.99	32.26	$C_{18}H_{16}N_8$	62.79	4.65	32.56
XXIII	36	201–203	62.01	5.59	32.17	$C_{18}H_{18}N_8$	62.43	5.20	32.37

2-aminopyrimidine (**VII**) (34300 cm^{-1}). At the same time in the solution spectra of compounds with an azomethine bond the longwave maximum is displaced to higher frequencies due to extension of the conjugation system: 32100 and 30250 cm^{-1} for bases **XI, XII** respectively in contrast to the spectra of the initial azines containing absorption bands at 34800 (**V**) and 34200 cm^{-1} (**VI**) [15].

In the ^1H NMR spectra of bases **XII, XIII, VIII–XI** the signals of NH_2 groups present in the original 3-aminopyridine (**VI**) (3.69 ppm) and 2-aminopyridine (**V**) (5.85 ppm) disappear, and instead arises a proton signal from the azomethine group in the region 8.53–9.09 ppm (Table 2) in agreement with the published data on the azomethines of azole series [8–10].

Note the downfield position of the signal from H_X proton in the spectra of compounds **XII** (8.38 ppm), **VIII** (8.34 ppm), **X** (8.1 ppm) (Table 1) as compared to the 2-substituted 1-vinylimidazoles (δ 6.6–7.0 ppm) [16–18]. The same shift was observed in the spectra of azomethines prepared from 2-aminobenzimidazoles and 5-amino-3-(2-pyrrolyl)pyrazoles, and it testifies to the participation of H_X atom in an intramolecular hydrogen bond with a nitrogen of the azomethine group [8–10]. This situation suggests prevalence of mutual *cis*-,*trans*-orientation of azomethine and vinyl groups (*E*-isomer, A, B).

In the ^1H NMR spectra of azomethines prepared with the use of 2-aminopyridine, **XII, XIII**, the signal of azomethine proton H_{az} is observed downfield (δ 9.05, 9.09 ppm) compared to the signal of similar proton in the spectra of Schiff bases **VIII, IX** where the amine moiety originates from 3-aminopyridine (**VI**) (H_{az} δ 8.53 ppm) (Table 2). The spatial arrangement of molecules **XII, XIII** is also compatible with the presence of second intermolecular hydrogen bond between the proton of CH_{az}=N bond and endocyclic nitrogen of pyridine ring A; this suggestion rationalizes the large δ value of azomethine proton in these compounds.

In the ^1H NMR spectra of aminals **XX–XXIII, XVIII, XIX** the signals of NH_2 groups observed in the original 2-amnopyrimidine (**VII**) at 6,56 ppm and in 2-aminopyridine (**V**) at 5.85 ppm are lacking, and signals appear belonging to NH (δ 6.00–7.58 ppm) and CH (δ 7.27–7.72 ppm) groups (Table 3). These signals were distinguished by recording spectra in DMSO at 80°C, therewith the NH signals sifted upfield by 0.3 ppm whereas the CH signals did not move.

EXPERIMENTAL

^1H NMR spectra were registered on spectrometer Bruker DPX-400 at 20–25 and 70–80°C from solutions in DMSO-d_6 and $CDCl_3$, internal reference HMDS. IR spectra were recorded on spectrometer Bruker IFS 25 from samples pelletized with KBr or as mulls in mineral oil. Mass spectra were measured on

Table 2. ^{1}H NMR spectra of azomethines **VIII–XIII** ($CDCl_3$), δ, ppm

Compd no.	H_X, d.d	H_A, d	H_B, d	H_{CH_2}, q	H_{CH_3}, t	H^4	H^5	H^6, m	H^7, m	H_{as}, s	$H^{2'}$, d	$H^{3'}$, m	$H^{4'}$	$H^{5'}$, m	$H^{6'}$
VIII	8.34	5.03	5.36			7.46 s	7.26 s			8.55	8.51		7.52 m	7.32	8.49 m
IX				4.61	1.48	7.24 s	7.11 s			8.53	8.50		7.50 m	7.32	8.49 m
X	8.10	5.38	5.65			–	–	–	–	8.76	8.58		7.50 m	7.35	8.54 m
XI				4.85	1.51	7.86 d	7.39 m	7.39	7.48	8.76	8.59		7.59 m	7.25	8.59 d.d
XII[a]	8.38	5.10	5.68			8.08 d	7.34 d			9.09		7.42	7.91 t.d	7.39	8.50 m
XIII[a]				4.58	1.33	7.54 s	7.22 s			9.05		7.32	7.84 t	7.29	8.48 d

[a] Spectra recorded in DMSO-d_6.

Table 3. ^{1}H NMR spectra of aminals **XVIII–XXIII** (DMSO-d_6), δ, ppm

Compd no.	H_X, m	H_A, d	H_B, d	H_{CH_2}, q	H_{CH_3}, t	H^4	H^5	H^6, m	H^7	H_{CH}	H_{NH}	$H^{3'}$, m	$H^{4'}$	$H^{5'}$	$H^{6'}$, d
XVIII[a]	7.42	5.25	5.59			7.19–7.34 m	7.19–7.34 m	7.19–7.34	7.19–7.34 m	7.66 d	6.21 d	6.55	7.54 m	6.55 m	8.08
XIX[a]				4.46	1.37	7.23–7.28 m	7.23–7.28 m	7.23–7.28	7.23–7.28 m	7.72 d	6.02 d	6.59	7.37 m	6.59 m	8.10
XX	7.31	4.86	5.42			7.63 d	6.91 d			7.30 m	7.49 d		8.32 d	6.69 t	8.32
XXI				4.07	1.23	7.14 d	6.80 d			7.27 t	7.39 d		8.31 d	6.66 t	8.31
XXII	7.35	5.22	5.62			7.74 d	7.27 m	7.27	7.74 d	7.46 t	7.66 t		8.34 d	6.71 t	8.34
XXIII				4.36	1.26	7.48 d	7.21 m	7.21	7.48 d	7.51 t	7.58 d		8.33 d	6.69 t	8.33

[a] Spectra recorded in $CDCl_3$.

GC-MS spectrometer LKB-2091 (BROMMA) with direct sample admission into the ion source (vaporizer temperature 250°C), ionizing energy 60 eV. Syntheses were carried out in anhydrous solvents.

3-(1-Vinylimidazol-2-yl)methyleneaminopyridine (VIII). To a melt of amine **VI** (0.3 g, 3.19 mmol) was added at stirring aldehyde **I** (0.4 g, 3.19 mmol) The reaction mixture was heated at ~95°C for 8 h with simultaneous distillation of liberating water at 10–15 mm Hg into a cooled trap. The solid yellow substance obtained was ground with 50 ml of hexane. The precipitate obtained was filtered off, washed with hexane and ether, dried. Yield 0.5 g.

3-(1-Ethylimidazol-2-yl)methyleneaminopyridine (IX) was prepared in a similar way from 1.5 g (16 mmol) of amine **VI** and 2 g (16 mmol) of aldehyde **II** within 16 h. Yield 2.3 g.

3-(1-Vinylbenzimidazol-2-yl)hydroxymethyleneaminopyridine (Xa). To a solution of 0.54 g (3.19 mmol) of aldehyde **III** in 5 ml of benzene was added dropwise at stirring a solution of 0.3 g (3.19 mmol) of pyridine **VI** in 5 ml of benzene. The reaction mixture with the precipitate which separated within 1 min was stirred at room temperature for 2 h. The precipitate was filtered off, washed with benzene and ether, and dried. Yield 0.7 g.

3-(1-Ethylbenzimidazol-2-yl)methyleneaminopyridine (XI). To a solution of 0.56 g (3.19 mmol) of aldehyde **IV** in 10 ml of benzene was added by portions 0.3 g (3.19 mmol) of amine **VI**. The reaction mixture was boiled for 24 h, the solvent was evaporated till the volume of the mixture was 5 ml, and it was cooled. The yellow solution formed was poured into 20 ml of ether, the precipitate was filtered off and dried. Yield 0.5 g.

2-(1-Vinylimidazol-2-yl)methyleneaminopyridine (XII). To a melt of amine **V** (1.93 g, 20.5 mmol) was added aldehyde **I** (2.5 g, 20.5 mmol). The reaction mixture was heated at ~95°C for 12 h with simultaneous distillation of liberating water into a cooled trap. Then the residue was subjected to vacuum distillation. Yield 1.83 g.

2-(1-Ethylimidazol-2-yl)methyleneaminopyridine (XIII) was likewise prepared from a mixture of 1.93 g (20.5 mmol) of amine **V** and 2.5 g (20.5 mmol) of carbaldehyde **II**. Yield 2.6 g.

2-(1-Vinylbenzimidazol-2-yl)methylenediaminopyridine (XVIII). To a solution of 0.55 g (3.19 mmol) of aldehyde **III** in 20 ml of benzene was added at stirring by portions 0.6 g (6.38 mmol) of amine **V**. The reaction mixture was boiled for 26 h in a flask connected to the Dean-Stark trap. After evaporation to a volume of 10 ml the reaction mixture was poured into 30 ml of ether, the precipitate formed was washed with cold benzene and dried. Yield 0.75 g.

2-(1-Ethylbenzimidazol-2-yl)methylenediaminopyridine (XIX) was similarly synthesized from a mixture of 0.56 g (3.19 mmol) of aldehyde **IV** and 0.6 g (6.38 mmol) of amine **V**. Yield 0.67 g.

2-(1-Vinylimidazol-2-yl)methylenediaminopyrimidine (XX). To a solution of 2.57 g (21 mmol) of aldehyde **I** in 30 ml of ethanol was added at stirring 2 g (21 mmol) of amine **VII**. The reaction mixture was boiled for 20 h. Then the solvent was evaporated to a volume of 20 ml, the precipitate was filtered off, washed with ethanol and ether, and dried. Yield 1.75 g.

Likewise was synthesized **2-(1-ethylimidazol-2-yl)-methylenediaminopyrimidine (XXI)** from a mixture of 2.0 g (16 mmol) of aldehyde **II** and 1.53 g (16 mmol) of amine **VII**. The precipitate was filtered off, washed with acetone and ether. Yield 1.8 g, *m/z* 296.

Likewise was prepared **2-(1-vinylbenzimidazol-2-yl)methylenediaminopyrimidine (XXII)** from a mixture of 3.6 g (21 mmol) of aldehyde **III** and 2 g (21 mmol) of amine **VII** within 24 h. Yield 0.9 g.

Likewise was prepared **2-(1-ethylbenzimidazol-2-yl)methylenediaminopyrimidine (XXIII)** from a mixture of 3.65 g (21 mmol) of aldehyde **IV** and 2 g (21 mmol) of amine **VII**. Yield 1.3 g.

The constants, yields, and elemental analyses of compounds synthesized are given in Table 1.

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