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DEARYLATION WITH AROMATIZATION ON CYCLOCONDENSATION OF 4-(DIMETHYL-AMINO)BENZALDEHYDE, 2-PHENACYLAZA-HETEROCYCLES, AND 1,3-[N,C]DINUCLEOPHILES

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Three-component cyclocondensation involving p-(dimethylamino)benzaldehyde, 2-phenacylazaheterocycle, and a 1,3-[N,C]-nucleophile (3,5-dimethoxyaniline, 6-amino-1,3-dimethylpyrimidine-2,4-dione, 1-amino-3-methyl-5-phenylpyrazole) in boiling acetic acid is accompanied by aromatization of the initially formed annelated 4-(p-dimethylaminophenyl)-3-hetaryl-2-phenyl-1,4-dihydropyridines, the direction of which, with splitting off the dimethylaminophenyl substituent or its retention, is determined by the basicity of the hetaryl residue and the structure of the second ring, constructed on the binucleophile. A possible reaction mechanism is discussed.

Keywords: aldehydes, anilines, benzimidazoles, benzothiazoles, imidazoles, pyrazoles, pyrazolo-[3,4-*b*]pyridines, pyrido[2,3-*d*]pyrimidines, pyrimidines, quinolines, aromatization, dearylation, Hantzsch reaction, selectivity.

The method of obtaining pyridines according to Hantzsch, based on the cyclocondensation of aldehydes, acetoacetic ester, and ammonia with subsequent oxidation of the resulting 1,4-dihydropyridine, has been developed by modifying the synthesis conditions and the nature of the initial reactants [1, 2]. The three-component interaction of aldehydes with various carbonylmethylene-reactive compounds and 1,3-[N,C]-dinucleophiles is used in the synthesis of 1,4-dihydropyridines [2], pyrazolo[3,4-*b*]quinolines [3], and pyrido[2,3-*d*]pyrimidines [4].

The indicated reaction does not proceed selectively when using formaldehyde, which hinders the synthesis of compounds unsubstituted at position 4 of the pyridine ring [5-7]. It is known that formaldehyde may be replaced by other aldehydes, since on heating the reaction mixture in acetic acid the resulting products with an alkyl(aryl)-substituted dihydropyridine ring are inclined to undergo aromatization as a result of dealkylation (or dearylation) [4, 8]. The merit of such a method of synthesis is the combination into one process of the preparation of the dihydropyridine compound and its subsequent aromatization without the use of an oxidizing agent. However reaction with each of the studied aldehydes, with the exception of 4-(dimethyl-amino)benzaldehyde (**1**) is distinguished by a long duration (48 h) and low selectivity (10-55% yields) [4].

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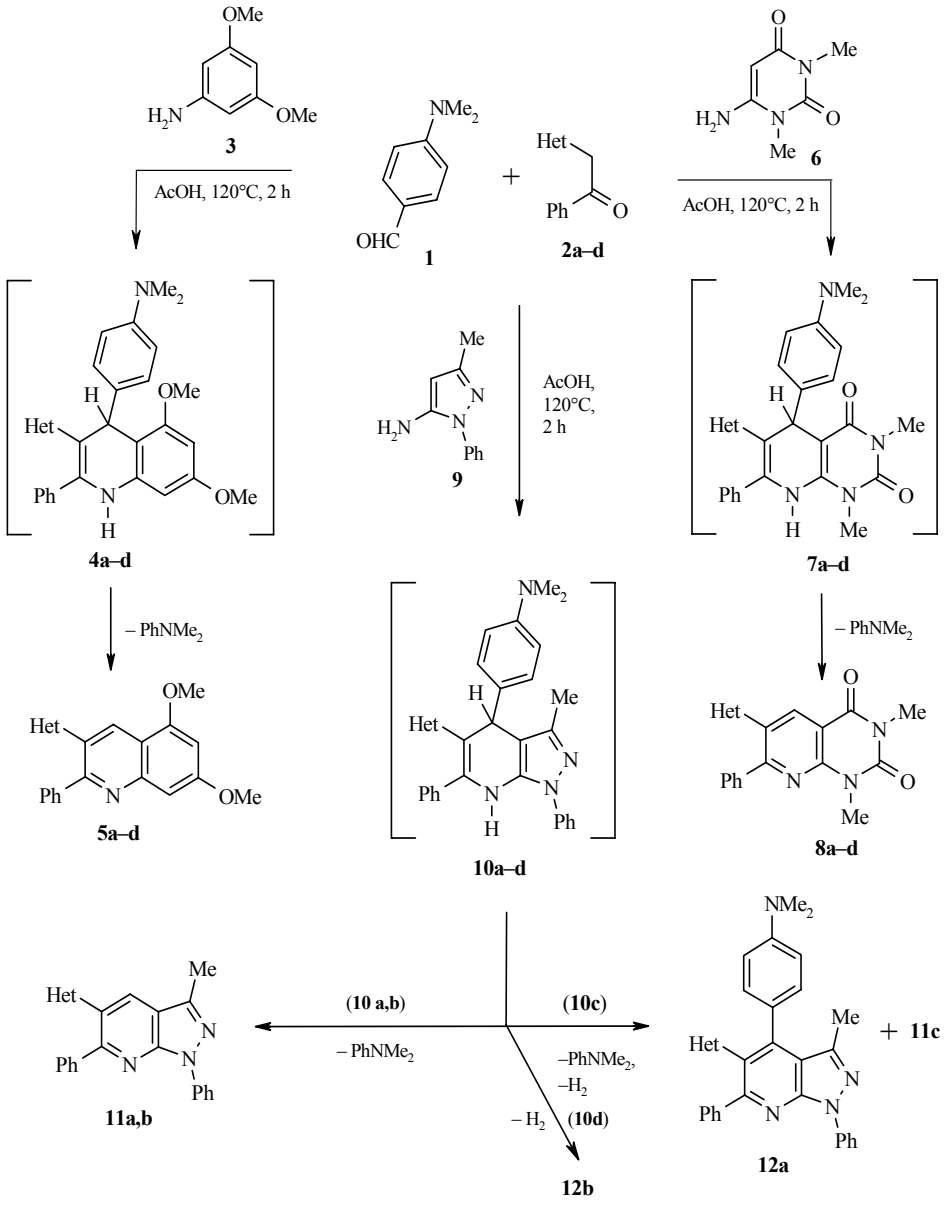
In the case of aldehyde **1** the formation of the corresponding compound containing a 1,4-dihydropyridine ring was smooth and accompanied selectively by aromatization as a result splitting off N,N-dimethylaniline [8]. On the basis of this a new approach was developed to the synthesis of compounds with no substituent in position 4 of the pyridine ring, used for obtaining derivatives of acridine [8], pyrazolo[3,4-*b*]pyridine [9], and pyrido-[2,3-*d*]pyrimidine [10].

The three-component interaction of aldehyde **1**, 2-phenacylazaheterocycles **2a-d**, and certain 1,3-[N,C]-dinucleophiles has been studied in the present work with the aim of extending the scope of the indicated method and of clarifying the mechanism of aromatization (see Scheme 1).

Scheme 1



2,4,5,7,8,10,11 a Het = 2-quinolyl, **b** Het = 2-benzothiazolyl;
2,4,5,7,8,10 c Het = 2-(1-methyl)benzimidazolyl, **d** Het = 2-imidazolyl;
12 a Het = 2-(1-methyl)benzimidazolyl, **b** Het = 2-imidazolyl

TABLE 1. Characteristics of the Synthesized Compounds

Compound	Empirical formula	Found, % / Calculated, %			mp, °C	Yield, %*
		C	H	N		
5a	$C_{26}H_{20}N_2O_2$	79.41 / 79.57	5.03 / 5.14	7.22 / 7.14	145.0-146.5	75
5b	$C_{24}H_{18}N_2O_2S$	72.19 / 72.34	4.38 / 4.55	6.89 / 7.03	183.5-185.0	81
5c	$C_{25}H_{21}N_3O_2$	75.79 / 75.93	5.18 / 5.35	10.47 / 10.63	198.5-200.0	73
5d	$C_{20}H_{17}N_3O_2$	72.33 / 72.49	5.23 / 5.17	12.47 / 12.68	269.5-271.0	62
8a	$C_{24}H_{18}N_4O_2$	72.94 / 73.08	4.68 / 4.60	14.12 / 14.20	233.0-234.5	80
8b	$C_{22}H_{16}N_4O_2S$	65.83 / 65.99	4.22 / 4.03	13.77 / 13.99	286.5-288.0	84
8c	$C_{23}H_{19}N_5O_2$	69.37 / 69.51	4.68 / 4.82	17.47 / 17.62	227.0-228.5	77
8d	$C_{18}H_{15}N_5O_2$	64.68 / 64.86	4.36 / 4.54	20.89 / 21.01	277.0-278.5	81
11a	$C_{28}H_{20}N_4$	81.38 / 81.53	4.78 / 4.89	13.37 / 13.58	153.0-154.5	75
11b	$C_{26}H_{18}N_4S$	74.48 / 74.62	4.18 / 4.34	13.22 / 13.39	191.5-193.0	90
12d	$C_{30}H_{26}N_6$	76.38 / 76.57	5.66 / 5.57	17.73 / 17.86	335.0-337.0	19

*Yields indicated are before purification by recrystallization.

We found that, as in the examples studied previously, the condensation of reactants **1**, **2a-d**, and 3,5-dimethoxyaniline (**3**), as the 1,3-dinucleophile, did not stop at the formation of compounds with a 1,4-dihydropyridine ring of type **4**, but was accompanied readily and selectively by the splitting off of N,N-dimethylaniline with the formation of the previously unknown quinoline derivatives **5a-d**.

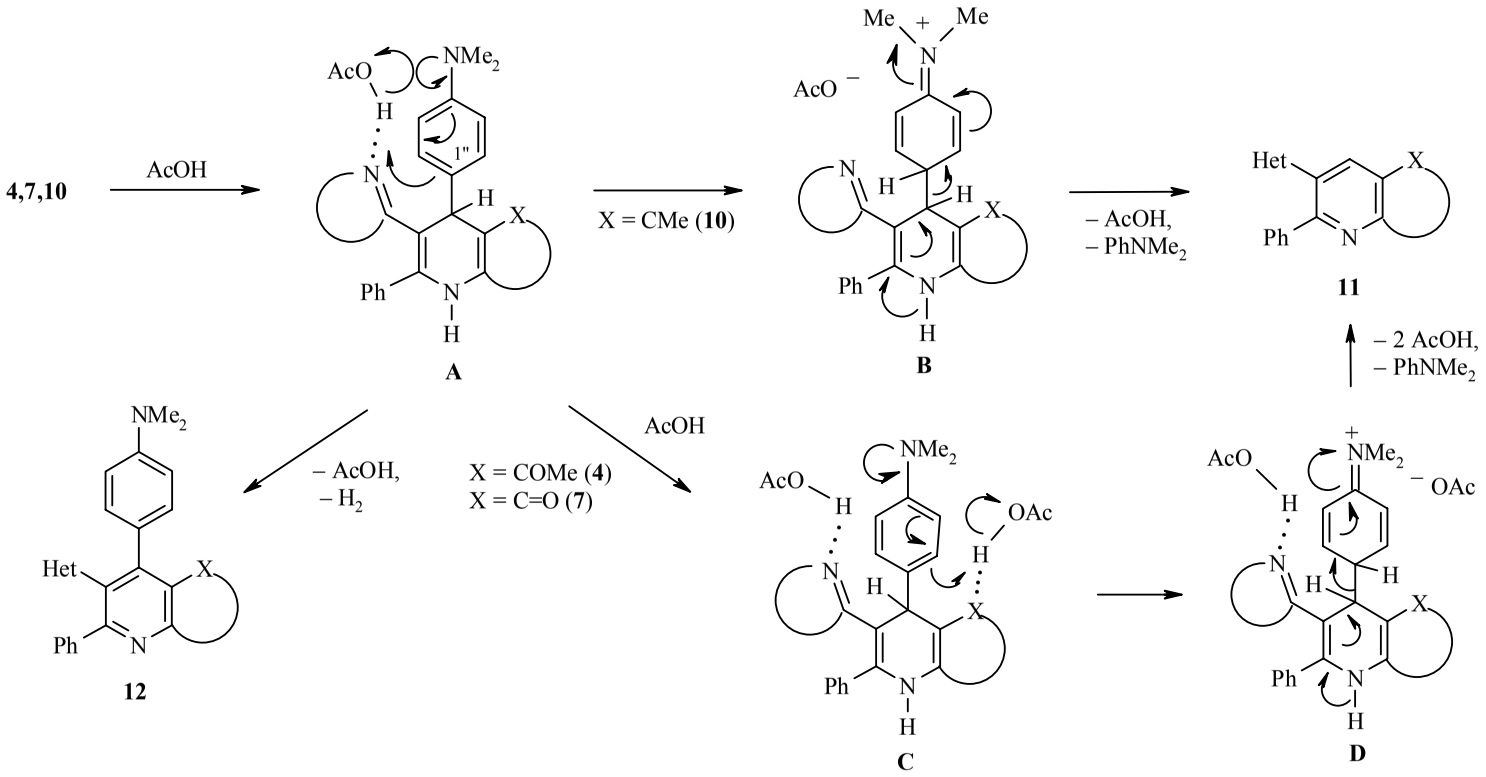
The reaction proceeds analogously in the case of 6-amino-1,3-dimethylpyrimidine-2,4-dione (**6**) through intermediates of type **7** and the subsequent formation of new pyrido[2,3-*d*]pyrimidine derivatives **8a-d**.

In the case of 5-amino-3-methyl-1-phenylpyrazole (**9**) the direction of aromatization of the cyclocondensation products depends on the nature of the initial 2-phenacylazaheterocycle **2**. With the derivatives of quinoline and benzothiazole **2a,b**, as in the examples given above, pyrazolo[3,4-*b*]pyridines **11a,b** are formed from products **10a,b**. On the other hand, when using derivatives of 1-methylbenzimidazole and imidazole **2c,d** aromatization of products **10c,d** takes place with retention of the (dimethylamino)phenyl substituent. In the case of reactant **2c** a mixture is formed of product **11c,** unsubstituted at position 4**,** and its 4-(*p*-dimethylaminophenyl) derivative **12a** in a molar ratio of 3:1 (according to data of ^{1}H NMR), which we were unable to separate. On using reactant **2d** only the (dimethylamino)phenyl-substituted product **12b** was successfully isolated from the reaction mixture in 19% yield.

The obtained results indicate that the the direction of aromatization of the intermediates **4**, **7**, and **10** is determined by the structure of the ring condensed with the dihydropyridine ring, and the basicity of the Het substituent. Reaction with retention of the $C_6H_4NMe_2$ group occurs only in the case of compounds **10c,d**, having a substituted pyrazole ring and the most basic Het substituents, 2-(1-methyl)benzimidazolyl and 2-imidazolyl respectively.

Analysis of the data given above permits expression of a possible mechanism for aromatization. The bonding of the nitrogen atom of the 2-azahetaryl fragment of compounds **4**, **7**, and **10** with acetic acid *via* a hydrogen bond leads to an adduct represented in Scheme 2 by the generalized formula **A**. In the case of X = CMe a proton of the acetic acid of this adduct may at enhanced temperatures protonate the electron-rich C-1"

Scheme 2


4,7,10
AcOH
AcO
NMe2
H
N
1"
H
X
Ph
N
H
A
X = CMe (10)
Me
+
N
Me
AcO −
B
– AcOH,
– PhNMe2
Het
X
Ph
N
11
– 2 AcOH,
– PhNMe2
– AcOH,
– H2
X = COMe (4)
X = C=O (7)
AcOH
NMe2
Het
X
Ph
N
12
OAc
C
+
NMe2
– OAc
D

atom of the dimethylaminophenyl substituent located nearby with transfer of a positive charge to the dimethylamino group and the formation of salt **B**. Subsequently, as a result of fission of proton from the nitrogen atom of the dihydropyridine ring and synchronous transfer of the negative charge from it to the NMe_2^+ group, it readily undergoes aromatization with simultaneous splitting off dimethylaniline and formation of the final product **11**. However if the Het substituent in adduct A possesses enhanced basicity then the ability to transfer proton from the nitrogen atom to an oxygen atom is reduced. In such a case aromatization is also possible due to air-oxidation or disproportionation, which leads to the formation of a product of type **12**.

TABLE 2. Data of ^{1}H NMR Spectra of the Synthesized Compounds

Com-pound	Chemical shifts, δ, ppm, SSCC (*J*, Hz)
5a	3.95 (3H, s, 5-OCH_3); 4.00 (3H, s, 7-OCH_3); 6.74 (1H, d, *J* = 1.8, H-6); 7.07-7.10 (2H, m, H-8,3'*); 7.17-7.33 (3H, m, 3H-*m*, -*p* Ph); 7.38 (2H, d, *J* = 7.5, 2H-*o* Ph); 7.60 (1H, m, H-6'); 7.78 (1H, m, H-7'); 7.91 (1H, d, *J* = 8.1, H-5'); 8.06 (1H, d, *J* = 8.1, H-4'); 8.10 (1H, d, *J* = 8.4, H-8'); 8.71 (1H,s, H-4)
5b	3.95 (3H, s, 5-OCH_3); 4.03 (3H, s, 7-OCH_3); 6.76 (1H, d, *J* = 1.8, H-6); 7.08 (1H, d, *J* = 1.8, H-8); 7.37-7.51 (7H, m, C_6H_5, H-5',6'); 7.96 (1H, d, *J* = 7.8, H-7'); 8.02 (1H, d, *J* = 7.8, H-4'); 8.98 (1H, s, H-4)
5c	3.10 (3H, s, NCH_3); 3.98 (3H, s, 5-OCH_3); 4.00 (3H, s, 7-OCH_3); 6.78 (1H, d, *J* = 1.8, H-6); 7.15 (1H, d, *J* = 1.8, H-8); 7.23-7.36 (5H, m, H-5',6', 3H-*m*, -*p* Ph); 7.42 (3H, m, 2H-*o* Ph, H-7'); 7.68-7.72 (1H, m, H-4'); 8.62 (1H, s, H-4)
5d	3.94 (3H, s, 5-OCH_3); 4.00 (3H, s, 7-OCH_3); 6.71 (1H, d, *J* = 1.8, H-6); 6.97 (1H, s, H-5'); 7.06 (1H, d, *J* = 1.8, H-8); 7.11 (1H, s, H-4'); 7.32-7.35 (3H, m, 3H-*m*, -*p* Ph); 7.38 (2H, d, *J* = 8.1, 2H-*o* Ph); 8.49 (1H, s, H-4); 11.91 (1H, s, NH)
8a	3.36 (3H, s, 1-CH_3); 3.66 (3H, s, 3-CH_3); 7.05 (1H, d, *J* = 8.1, H-3'); 7.32 (2H, t, *J* = 6.9, 2H-*m* Ph); 7.38-7.42 (3H, m, 3H-*o*, -*p* Ph); 7.62 (1H, m, H-6'); 7.79 (1H, m, H-7'); 7.93 (1H, d, *J* = 8.1, H-5'); 8.05 (1H, d, *J* = 8.1, H-4'); 8.14 (1H, d, *J* = 8.7, H-8'); 8.62 (1H, s, H-5)
8b	3.37 (3H, s, 1-CH_3); 3.64 (3H, s, 3-CH_3); 7.39-7.57 (7H, m, C_6H_5, H-5',6'); 7.97 (1H, d, *J* = 7.8, H-7'); 8.03 (1H, d, *J* = 8.1, H-4'); 8.86 (1H, s, H-5)
8c	3.12 (3H, s, 1'-CH_3); 3.35 (3H, s, 1-CH_3); 3.70 (3H, s, 3-CH_3); 7.24-7.27 (2H, m, H-5',6'); 7.31 (2H, t, *J* = 6.9, 2H-*m* Ph); 7.35-7.45 (4H, m, 3H-*o*, -*p* Ph, H-7'); 7.68-7.72 (1H, m, H-4'); 8.53 (1H, s, H-5)
8d	3.33 (3H, s, 1-CH_3); 3.62 (3H, s, 3-CH_3); 6.98 (1H, s, H-5'); 7.15 (1H, s, H-4'); 7.33-7.42 (5H, m, C_6H_5); 8.41 (1H, s, H-5); 12.08 (1H, s, NH)
11a	2.70 (3H, s, 3-CH_3); 7.15 (1H, d, *J* = 8.1, H-3'); 7.31-7.35 (4H, m, 3H-*m*, -*p* CPh, H-*p* NPh); 7.42-7.44 (2H, m, 2H-*o* CPh); 7.55-7.60 (2H, m, 2H-*m* NPh); 7.63 (1H, m, H-6'); 7.80 (1H, m, H-7'); 7.95 (1H, d, *J* = 7.8, H-5'); 8.07 (1H, d, *J* = 8.1, H-4'); 8.15 (1H, d, *J* = 8.7, H-8'); 8.38 (2H, d, *J* = 7.5, 2H-*o* NPh); 8.68 (1H, s, H-4)
11b	2.71 (3H, s, 3-CH_3); 7.41-7.59 (9H, m, 4-C_6H_5, 3H-*m*, -*p* NPh, H-5',6'); 7.98-8.05 (2H, m, H-4',7'); 8.30 (2H, d, *J* = 8.2, 2H-*o* NPh); 8.89 (1H, s, H-4)
11c	2.68 (3H, s, 3-CH_3); 3.14 (3H, s, 1'-CH_3); 7.25-7.37 (6H, m, H-5',6', H-*p* NPh, 3H-*m*, -*p* CPh); 7.43 (3H, m, 2H-*o* CPh, H-7'); 7.58 (2H, t, *J* = 7.8, 2H-*m* NPh); 7.68-7.70 (1H, m, H-4'); 8.37 (2H, d, *J* = 7.5, 2H-*o* NPh); 8.70 (1H, s, H-4)
12a	2.13 (3H, s, 3-CH_3); 2.84 (6H, s, $N(CH_3)_2$); 3.23 (3H, s, 1'-CH_3); 6.59 (2H, d, *J* = 7.8, 2H-*m* Ar*[2]); 7.13-7.22 (7H, m, H-5',6', 2H-*o* Ar*[2], 3H-*m*, -*p* CPh); 7.32-7.39 (4H, m, 2H-*o* CPh, H-*p* NPh, H-7'); 7.53-7.58 (3H, m, 2H-*m* NPh, H-4'); 8.34 (2H, d, *J* = 7.5, 2H-*o* NPh)
12b	2.09 (3H, s, 3-CH_3); 2.91 (6H, s, $N(CH_3)_2$); 6.63 and 7.08 (2H and 2H, two d, *J* = 7.8, Ar*[2]); 6.77 and 6.88 (1H and 1H, two s, H-4' and H-5'); 7.27-7.33 (4H, m, 3H-*m*, -*p* CPh, H-*p* NPh); 7.38-7.40 (2H, m, 2H-*o* CPh); 7.56 (2H, t, *J* = 7.8, 2H-*m* NPh); 8.33 (2H, d, *J* = 7.8, 2H-*o* NPh); 11.72 (1H, s, NH)

* Here and subsequently the position of the proton in the Het substituent is marked with a prime.

*[2] Ar = $C_6H_4N(CH_3)_2$-*p*.

If X = COMe or C=O then binding of the oxygen atom of these fragments with acetic acid by an intermolecular hydrogen bond and the formation of adduct **C** is fully possible. In the latter the process of proton transfer to atom C-1" may occur analogously to that considered above or through the formation of the solvated salt **D**. Subsequent aromatization with splitting off dimethylaniline and decomposition of the acetic acid adduct (on diluting the reaction mixture with water) leads to a product of type **11**. In this case the enhanced basicity of the Het substituent is not a hindrance to the splitting off dimethylaniline, but its reduced basicity and the presence of the oxygen-containing fragment X are correspondingly especially favorable.

The phenomenon of accelerating reactions under the influence of neighboring groups is known under the name synarthetic (or anchimeric) acceleration [11]. In the actual case it is evident that C-protonation at position 1" of the 4-(dimethylamino)phenyl substituent without the participation of neighboring functional groups must experience significant difficulty, since the direct approach of proton to the reaction center is blocked by significant steric hindrance.

The specific behavior of aldehyde **1** in the Hantzsch reaction is, very probably, not peculiar to it alone. Similar behavior may be expected for aldehydes in which the formyl group is under a powerful electron-donating influence. The latter takes place, for example, in 4-(dialkylamino)benzaldehydes, pyrrolecarbaldehydes, 3-indolecarbaldehydes, etc. The clear advantage of compound **1** over others is its availability.

The composition and structure of the obtained compounds are in accordance with the data of elemental analysis (Table 1) and ^{1}H NMR spectra (Table 2).

Three-component condensation involving dimethylaminobenzaldehyde has been studied on new examples in the present work. The results obtained show the possibility of two routes for aromatization of the initially formed products with a *p*-dimethylaminophenyl-substituted dihydropyridine ring (with and without splitting off dimethylaniline) and enable factors affecting the direction of this process to be clarified.

EXPERIMENTAL

A check on the progress of reactions and the purity of the compounds synthesized was carried out by TLC on Silufol UV-254 plates in the solvent system benzene–ethanol, 9:1, with visualization in UV light. The ^{1}H NMR spectra of compounds were recorded on a Varian VXR-300 (300 MHz) in DMSO-d_6, standard was TMS. All the compounds synthesized were dried for 5 h at 125^{o}C before carrying out elemental analysis and spectral investigations.

Cyclocondensation of 4-(Dimethylamino)benzaldehyde (1), 2-(Phenacylazaheterocycles 2a-d and 3,5-Dimethoxyaniline (3) or Heterocyclic Amines 6, 9 (General Method). A mixture of aldehyde **1** (10 mmol), compound **2** (10 mmol), amine **3** (1.5 mmol), or amine **6, 9** (1.0 mmol), and glacial acetic acid (2 ml) (in the case of compound **2d** 1 ml acid was used) was maintained at 120°C for 2 h. The methods of processing the cooled reaction mixture, isolating, and purifying the products are given below.

5,7-Dimethoxy-2-phenyl-2',3-biquinoline (5a) was obtained from compounds **1**, **2a**, and **3**. Water (8 ml) was added to the reaction mass and the mixture heated to boiling with stirring. After cooling, the solidified oil was triturated to a powder, which was filtered off, washed with water, dried, and dissolved in the minimum quantity of benzene. The solution obtained was applied to a column of aluminum oxide (neutral) and eluted with benzene, collecting the light-yellow fraction, which was then evaporated to dryness. The residue was dissolved with heating in cyclohexane (10 ml), hexane (5 ml) was added to the solution, and the mixture maintained at 20°C for 4 h, and at 0^{o}C for 1h. The separated solid product **5a** was filtered off, and washed with hexane.

3-(1,3-Benzothiazol-2-yl)-5,7-dimethoxy-2-phenylquinoline (5b) was obtained from compounds **1**, **2b**, and **3**. Water (2 ml) was added to the reaction mass, which was boiled with stirring until the start of

crystallization. After cooling, the solid product **5b** was filtered off, washed with a mixture of 2-propanol–water, 1:1, dried, and recrystallized from a mixture of pyridine–water, 1:1.

5,7-Dimethoxy-3-(1-methyl-1H-benzimidazol-2-yl)-2-phenylquinoline (5c) was obtained from compounds **1**, **2c**, and **3**. Water (8 ml) was added to the reaction mass and the mixture heated to boiling with stirring. After cooling, the solid product **5c** was triturated, filtered off, washed with a mixture of 2-propanol–water, 1:1, dried, and recrystallized from a mixture of pyridine–water, 3:2.

3-(1H-Imidazol-2-yl)-5,7-dimethoxy-2-phenylquinoline (5d) was obtained from compounds **1**, **2d**, and **3** in glacial acetic acid (1 ml). As in the procedure given above for compound **5c** the reaction mass was treated with water (4 ml), and product **5d** isolated, and purified, then recrystallized from a mixture of pyridine–water, 1:1.

1,3-Dimethyl-7-phenyl-6-quinol-2-ylpyrido[2,3-*d*]pyrimidine-2,4-(1H,3H)-dione (8a) was obtained from compounds **1**, **2a**, and **6**. As in the procedure given for compound **5b** the reaction mass was treated with water (2 ml). Product **8a** was isolated, washed, and dried, then recrystallized from a mixture of pyridine–water, 2:1.

6-(1,3-Benzothiazol-2-yl)-1,3-dimethyl-7-phenylpyrido[2,3-*d*]pyrimidine-2,4-(1H,3H)-dione (8b) was obtained from compounds **1**, **2b**, and **6** as for **8a**.

1,3-Dimethyl-6-(1-methyl-1H-benzimidazol-2-yl)-7-phenylpyrido[2,3-*d*]pyrimidine-2,4-(1H,3H)-dione (8c) and 6-(1H-Imidazol-2-yl)-1,3-dimethyl-7-phenylpyrido[2,3-*d*]pyrimidine-2,4-(1H,3H)-dione (8d) were obtained from compounds **1**, **2c**, and **6** in acetic acid (2 ml) and from compounds **1**, **2d**, and **6** in acetic acid (1 ml) respectively. In the first case the reaction mass was treated with water (8 ml) and in the second with water (4 ml). In the rest of the treatment the isolation and purification of products **8c,d** were analogous to that indicated for compound **8a**.

2-(3-Methyl-1,6-diphenyl-1H-pyrazolo[3.4-*b*]pyrid-5-yl)quinoline (11a) was obtained from compounds **1**, **2a**, and **9**. Water (8 ml) was added to the reaction mass and the mixture heated to boiling with stirring. The cooled mass was filtered, the solid washed with water, dried, and dissolved in the minimum amount of methylene chloride. The solution was applied to a column of aluminum oxide (neutral), eluted with diethyl ether, collecting the first colorless fraction, which was evaporated to dryness. The residue, product **11a**, was triturated to a powder, and recrystallized from a mixture of 2-propanol–water, 5:1.

5-(1,3-Benzothiazol-2-yl)-3-methyl-1,6-diphenyl-1H-pyrazolo[3,4-*b*]pyridine (11b) was obtained from compounds **1**, **2b**, and **9**. The reaction mass was treated by the procedure given for compound **5b**. The isolated solid product **11b** was recrystallized from toluene.

3-Methyl-5-(1-methyl-1H-benzimidazol-2-yl)-1,6-diphenyl-1H-pyrazolo[3,4-*b*]pyridine (11c) and 3-Methyl-4-[4-(dimethylamino)phenyl]-5-(1-methyl-1H-benzimidazol-2-yl)-1,6-diphenyl-1H-pyrazolo-[3,4-*b*]pyridine (12a) were obtained from compounds **1**, **2c**, and **9**. Water (8 ml) was added to the reaction mass and the mixture was heated to boiling with stirring. After cooling solid-1 was filtered off, washed with water, dried, and boiled with stirring in acetonitrile (2 ml) for 2-3 min. Solid-2, isolated after cooling, was filtered off, and washed with acetonitrile. Solid-2 was a mixture of products **11c** and **12a** in a molar ratio of 3 : 1 (according to data of ^{1}H NMR), its mp 196-198^{o}C was unchanged after crystallization from a mixture of pyridine–water, 2:1.

4-[4-(Dimethylamino)phenyl]-5-(1H-imidazol-2-yl-3-methyl-1,6-diphenyl-1H-pyrazolo[3,4-*b*]pyridine (12b) was obtained from compounds **1**, **2d**, and **9**. As in the procedure given for compound **5b** the reaction mass was treated with water (2 ml), the solid filtered off, washed, and dried, to give the solid product **12b**, which was recrystallized from a mixture of pyridine–water, 2:1.

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