STRUCTURE AND REACTIVITY OF 2-METHYL-9-HYDROXY-4*H*-PYRIDO[1,2-*a*]PYRIMIDIN-4-ONE

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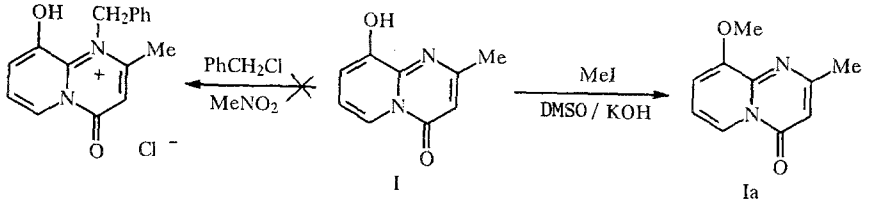
It has been shown that the alkylation of 2-methyl-9-hydroxy-4H-pyrido[1,2-a]pyrimidin-4-one takes place at the oxygen atom, but electrophilic substitution takes place mainly at position 8 of the molecule (the ortho position relative to the hydroxy group).

Continuing many years of work on the synthesis and studies of structure and reactivity of nitrogen heterocycles containing a phenolic hydroxyl in the β-position relative to the ring nitrogen atom, we have focused our attention on the structure and electrophilic substitution reactions of 2-methyl-9-hydroxy-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (I) [1], a compound that is of undoubted interest in the synthesis of physiologically active compounds with a broad spectrum of action [2].

In the initial stage, we studied the alkylation of the 9-hydroxypyridopyrimidinone I with the aim of obtaining a fixed structure of this compound, of the type of Ia, so that it would be possible to use UV spectroscopy to study the equilibrium between the neutral and ionic forms of the 9-hydroxypyridopyrimidine.

The alkylation of 9-hydroxy-4*H*-pyrido[1,2-*a*]pyrimidin-4-one I was accomplished successfully by extended heating with excess methyl iodide in DMSO in the presence of KOH. It was established, on the basis of data obtained by various physicochemical methods, that this reaction forms the *O*-methylated product Ia. In the PMR spectrum (in DMSO-d_6) of the compound Ia that we obtained, a singlet appears from protons of the OCH_3 group at 3.95 ppm. In the mass spectrum, a peak is observed for the molecular ion $M^{+\cdot}$ 190. The yield of compound Ia was no greater than 10%. Similar results were obtained by methylation of the pyridopyrimidinone I in the presence of sodium hydride. Attempts to obtain the product of *N*-alkylation by extended heating of the pyridopyrimidinone I with benzyl chloride in nitromethane were unsuccessful; only the original compound I was recovered from the reaction mass.



Among the reactions of electrophilic substitution, we studied aminomethylation, nitration, halogenation, and azo coupling. It had been shown previously that electrophilic substitution reactions, in particular nitration, are directed at position 3 of the pyridopyrimidine system [3]. It was of interest to determine how the introduction of the hydroxy group at position 9 would influence the orientation of substitution and the reactivity of the two-ring system.

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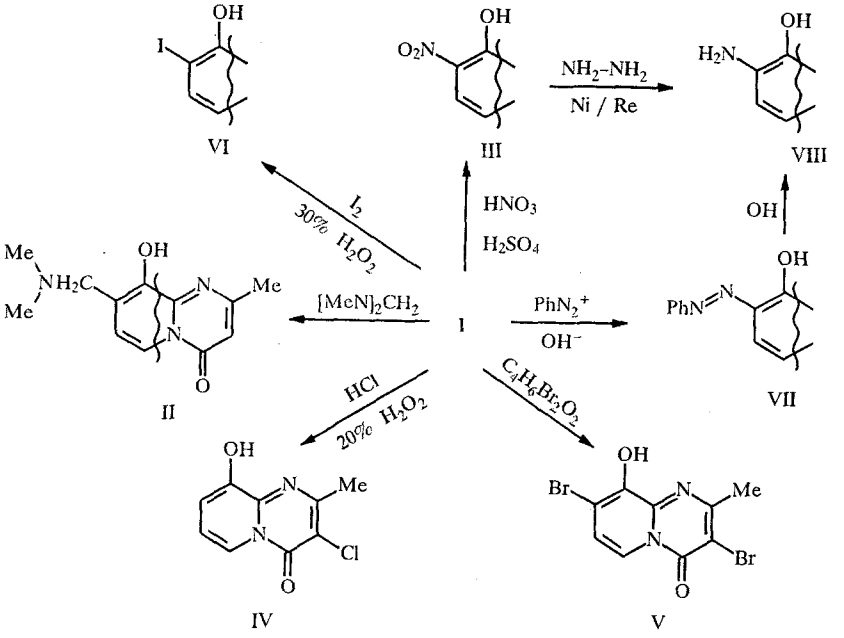
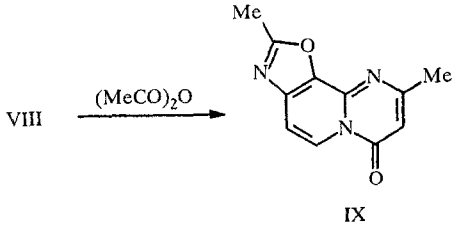
For the synthesis of the 8-dimethylaminomethyl derivative II, we used as the aminomethylating agent *N,N,N',N'*-tetramethylmethylenediamine [4]. Upon heating this compound with the pyridopyrimidinone I in benzene, we obtained only the Mannich base II, monosubstituted in position 8.

Upon nitration of compound I with a mixture of concentrated HNO_3 and H_2SO_4 [5], apparently involving participation of the protonated form in the reaction, we noted the formation of the nitro derivative III, but could not find any trace of the dinitro derivative. Reduction of the nitro compound gives the known compound 8-amino-9-hydroxypyridopyrimidin-4-one VIII [6]. This means that the nitration takes place in position 8.



In contrast to the reactions of aminomethylation and nitration, we find that when the pyridopyrimidinone I is chlorinated by the method of [4], the substitution takes place at position 3 of the pyrimidine ring, forming the monochloro derivative IV. Bromination of compound I by dioxane dibromide in acetic acid gives the dibromo derivative V. Attempts to obtain a monobromo-substituted pyridopyrimidinone by interaction of compound I with dioxane dibromide or with bromine in acetic acid were not successful. Upon reaction of the pyridopyrimidinone I with an alcohol solution of iodine in the presence of hydrogen peroxide, substitution takes place at position 8 of the two-ring system, forming the monoiodo derivative VI. Thus, the halogenation of compound I may go in different directions, depending on the conditions of the reaction.

Our experiments on azo coupling of compound I were performed in an alkaline media by a method given in [8]. The resulting azo compound VII was reduced to the 8-amino derivative VIII, the structure of which was established by means of PMR spectroscopy. The structure of compound VIII was confirmed by condensation with acetic anhydride, giving a new compound with an oxazole ring IX.



Thus, we can say that the preferred position for electrophilic substitution of the pyridopyrimidinone I is position 8, apparently because of the presence of the hydroxy group in the *ortho* position.

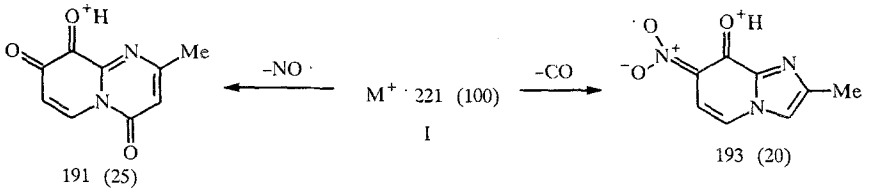
The structure of the synthesized compounds I—IX was confirmed by mass spectra with fast-atom (argon) bombardment in a glycerol matrix or with electron impact as the ion source (Table 1). With fast-atom bombardment of the pyridopyrimidinones I, Ia, II, IV—VI, VIII, and IX, intense peaks (up to 100%) of quasimolecular ions $[M + H]^+$ are registered. In the case of compounds II and IV—VI, formation of protonated fragment ions is observed, as a result of

TABLE 1. Characteristics of Derivatives of 2-Methyl-9-hydroxy-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (Ia—IX)

Compound	mp, °C	Mass spectra	
		electron impact, mz (I_{rel}, %)	Ar bombardment, m/z (I_{rel}, %)
Ia	150...151	$M^{+\cdot}$: 190	$[M+H]^+$: 191 (100)
II	148...150	$M^{+\cdot}$: 233 (23), 218 (12), 190 (100), 176 (5), 162 (33)	$[M+H]^+$: 234 (20), 218 (4), 189 (100), 173 (5), 161 (6)
III	300...301	$M^{+\cdot}$: 221 (100), 191 (25), 193 (20)	—
IV	189...190	$M^{+\cdot}$: 212 (35), 210 (100), 184 (12), 182 (36), 156 (5), 154 (15), 147 (15), 135 (20)	$[M+H]^+$: 213 (33), 211 (10), 177 (17)
V	233...235		$[M+H]^+$: 337 (4), 335 (6), 333 (4), 257 (20), 255 (20), 177 (100)
VI	173...175	$M^{+\cdot}$: 302 (< 2), 287 (8), 274 (100), 254 (12), 246 (15), 220 (12), 176 (35), 147 (35)	$[M+H]^+$: 303 (50), 177 (60), 165 (100)
VII	99...101		
VIII	220...222	$M^{+\cdot}$: 191 (100), 176 (5), 163 (35), 134 (10), 124 (14)	$[M+H]^+$: 192 (100)
IX	222...224		$[M+H]^+$: 216 (100)

decomposition or detachment of substituents. For example, in the spectrum of the pyridopyrimidinone II, along with the peak of the ion $[M + H]^+$ 234 (20),* signals are registered for the fragment ions $[M - CH_3]^+$ 218 (4) and $[M - N(CH_3)_2]^+$ 189 (100). For the halogen derivatives IV-VI, peaks are registered for $[M + H]^+$ and fragments $[M - n\text{Hal} + (n + 1)H]^+$ 177, where $n = 1$ for IV and VI, or $n = 2$ for V. It must be noted that the relative intensities of these peaks are highly dependent on the nature of the halogen (mass, electronegativity) and also on the chemical stability of the molecules of IV—VI as a whole. The behavior of the halogen-substituted pyridopyrimidinones in the glycerol matrix upon bombardment by fast argon atoms is also greatly dependent on intermolecular chemical processes with active participation of the glycerol molecules [9].

A characteristic feature of the mass spectra of the pyridopyrimidinones I—IV and VI under conditions of electron impact is the ejection of the CO group of the pyrimidine ring, followed by contraction to give a five-membered ring. In the spectra of these compounds we also observe peaks of the ions $[M - 200]^{+\cdot}$ with a lower intensity, the formation of which can be explained by the subsequent contraction of the pyridine ring to a five-membered ring. For the nitro derivative III, the peak of the molecular ion is registered, as well as peaks of typical fragment ions, the formation of which is due to rearrangements with participation of the nitro group and contraction of the pyrimidine ring as a result of ejection of the CO group.



The PMR spectra of the synthesized pyridopyrimidinones, in comparison with the spectrum of the unsubstituted 2-methyl-4*H*-pyrido[1,2-*a*]pyrimidin-4-one [10] when recorded under identical conditions, can be used for a clear-cut determination of the position of the substituents that have been introduced. The parameters of the spectra of these compounds are listed in Table 2. In the weak field of the PMR spectrum of the pyridopyrimidinone X (unsubstituted in position 9), which was synthesized by a method described previously [3], multiplet signals are registered from protons of the pyridine fragment. A weakly split doublet at 2.38 ppm with $^4J_3CH_3 = 0.5$ Hz pertains to methyl protons, and an unresolved quartet at 6.29 ppm pertains to the 3-H proton. Of the two multiplets from the 6-H and 9-H protons, which are characterized by the presence of only one vicinal constant ^3J, in the weaker field at 892 ppm we observe a signal from the 6-H hydrogen in the α-position

*Here and subsequently, values are given for m/z (and relative intensity, %).

TABLE 2. PMR Spectra of Derivatives of 2-Methyl-4*H*-pyrido[1,2-*a*]pyrimidin-4-one in DMSO-d_6

Compound	Chemical shifts (δ, ppm) and SSCCs (J, Hz)					
	$C_{(2)}$—CH_3	R^3	R^6	7-H	R^8	R^9
I*	2,40, s	6,28, s	8,43,dd, J_{67}=7,2, J_{68}=1,4	7,16, dd, J_{67}=7,2, J_{78}=7,5	7,21, dd, J_{68}=1,4, J_{78}=7,5	
Ia	2,37, s	6,31,s	8,52,dd, J_{67}=6,3, J_{68}=1,9	7,20,dd, J_{67}=6,3, J_{78}=7,6	7,34,dd, J_{68}=1,9, J_{78}=7,6	CH_3O, 3,95, s
II	2,40, s, $^4J_{3,CH3}$=0,5	6,24, q, ***, $^4J_{3,CH3}$=0,5	8,41,d, J_{67}=7,2	7,19,d, J_{67}=7,2	Me_2NCH_2, 2,26, s, 3,62,	
IV	2,57, s		8,44, m X part of AA'X system	7,25, m AA'-	7,25, m part of AA'X system	
V	2,62, s		8,28, d, J_{67}=7,8	7,45, d, J_{67}=7,8		
VI	2,48, s	6,22, s	8,05,d, J_{67}=7,7	7,40 d, J_{67}=7,7		
VIII	2,41, s	5,95, s	8,50,d, J_{67}=7,7	7,04 d, J_{67}=7,7	NH_2, 7,89, br	
IX	2,40, d, *** $^4J_{3,CH3}$=0,6	6,30, q, *** $^4J_{3,CH3}$=0,6	8,86, , J_{67}=7,4	7,60, , J_{67}=7,4		CH_3CO, 2,76,
X**	2,38, d, *** $^4J_{3,CH3}$=0,5	6,29, q, *** $^4J_{3,CH3}$=0,5	8,92, ddd, J_{67}=7,0, J_{68}=1,6, J_{69}=0,8	7,31, ddd, J_{67}=7,0, J_{78}=6,9, J_{79}=1,4	7,92, ddd, J_{68}=1,6, J_{78}=6,9, J_{89}=9,0	7,61, ddd, J_{69}=0,8, J_{79}=1,4, J_{89}=9,0

*Spectrum was recorded in a Bruker AC-250 spectrometer with a working frequency of 250 MHz.

**Values of chemical shift and SSCC are given on the basis of data from iteration of spectrum.

***Unresolved.

relative to the bridge nitrogen. The signal from the 9-H proton has a smaller value of the chemical shift (7.61 ppm). The multiplets of the 7-H and 8-H protons, which are characterized by the presence of two vicinal SSCCs, were differentiated by means of spectra of double resonance with 6-H, on the basis of disappearance of the ^3J constant in the signal at 7.31 ppm. An analysis of the vicinal SSCCs shows that $^3J_{89}$ = 9.0 Hz, substantially greater than the other two values: $^3J_{67}$ = 7.0 and $^3J_{78}$ = 6.9 Hz.

For the hydroxy derivative of the pyridopyrimidinone I, along with two broadened singlets at 2.40 and 6.28 ppm, which are assigned to methyl and methine protons of the pyrimidine fragment, in the weak field of the spectrum we observe multiplets from three protons of the pyridine ring. The signal at 7.16 ppm is characterized by two vicinal constants, indicating the presence of a substituent in position 6 or 9. The position of the signal that is the farthest downfield (8.43 ppm) enables us to assign this multiplet to the 6-H proton, i.e., to confirm beyond doubt the presence of the hydroxyl group in position 9 (not 6); and this is also evidenced by the relatively low values of the vicinal SSCCs, 7.2 and 7.5 Hz. A comparison of the spectra of compound X and its hydroxy derivative I shows that the introduction of the hydroxyl group is manifested in upfield shifts of the multiplets of the 8-H, 6-H, and 7-H protons amounting to 0.71, 0.49, and 0.15 ppm, respectively. This brings the 8-H and 7-H signals closer together (the difference of shifts amounting to 0.05 ppm) and complicates the spectrum recorded at 90 MHz (the AB part of the ABX spin system). For an accurate measurement of the chemical shift and the SSCC of the signals from the pyridine ring protons, the spectrum of compound I was recorded in a spectrometer with a working frequency of 250 MHz, with subsequent iteration. In the spectrum of the 9-methoxypyridopyrimidinone Ia, we observe the same sequence

in the positions of the signals from the methine protons as in the spectrum of compound I. The downfield shifts of the multiplets of the pyridine ring proton reflect the effect of *O*-methylation. A singlet of the methoxyl protons is registered at 3.95 ppm.

In the spectrum of the chlorine derivative IV, a narrow singlet of methyl protons is registered, and also two groups of multiplets at 8.44 ppm (1H, 6-H, X part of AA'X spin system) and 7.25 ppm (2H, 7-H and 8-H, AA' part of AA'X system). There is no signal of the 3H proton in the 6.2-6.3 ppm region. These data show that in compound IV, the chlorine is located on the $C_{(3)}$ carbon atom. The presence of halogen substituents at $C_{(8)}$ in the derivatives V and VI is confirmed by the presence, in the weak field of the spectra of these compounds, of doublet signals of 6-H protons (8.28 and 8.05 ppm) with vicinal constants. The doublets of the 7-H protons are farther upfield (7.45 and 7.40 ppm). The absence of any signal of the 3-H proton in the spectrum of the dibromo derivative V is an indication of the position of the second bromine atom, at the $C_{(3)}$ carbon.

In the spectrum of compound II, the presence of two doublet signals at 8.41 and 7.19 ppm and a vicinal constant 7.2 Hz — these signals assigned to 6-H and 7-H protons — indicates that the dimethylaminomethylene group is positioned at the $C_{(8)}$ carbon. Singlets of protons of the *N*-methyl and *N*-methylene groups are registered at 2.26 and 3.62 ppm. In the spectrum of the aminopyridopyrimidinone VIII, along with broadened signals of methyl and methine protons of the pyrimidine fragment, two doublets are registered at 8.50 and 7.04 ppm, and vicinal SSCC 7.7 Hz. The chemical shift observed for the downfield doublet enables us to assign it to the 6-H proton, so that the position of the amino group can be identified: at the $C_{(8)}$ carbon. The protons of the amino group are registered in the form of a broad signal at 7.89 ppm. A doublet of the 6-H proton at 8.86 ppm with a vicinal constant 7.4 Hz is observed in the spectrum of the product from the condensation of the 8-amino derivative VIII with acetic anhydride, i.e., compound IX. The singlet of the methyl group proton in the oxazole ring is registered at 2.76 ppm. In comparison with the spectrum of the original compound VIII, all of the signals of the methine protons in the molecule of IX are shifted downfield by 0.35—0.56 ppm, reflecting an effect of annelation of the pyridopyrimidinone system by the oxazole ring.

We used UV spectroscopy to measure the ionization constants of the pyridopyrimidinone I $pK_1 = 3.73$ (protonation) and $pK_2 = 7.65$ (deprotonation), and also the ionization constant of its *O*-methyl derivative Ia, pK = 3.49. The pK_a data and the UV spectra indicate that the molecule of I is a conventional ampholyte. The very similar values of pK_1 (I) and pK (Ia) indicate that in the molecule of I, the protonation takes place at a nitrogen atom, probably $N_{(1)}$. It is known that the equilibrium between the neutral and zwitterion forms of the molecules of hydroxy derivatives of pyridine is shifted, in dioxane, in the direction of the neutral form; and this brings about characteristic changes in the UV spectra [9]. At the same time, the UV spectra of compound I in aqueous dioxane solutions remain practically unchanged as the dioxane concentration is increased from 0 to 80%. This fact indicates preference for the neutral form of the molecule of the pyridopyrimidinone I in aqueous solutions.

EXPERIMENTAL

The mass spectra obtained by bombardment with fast atoms of argon in a glycerol matrix were obtained in an MI-1201É instrument. The energy of the argon atoms was 5 keV. The electron-impact mass spectra were obtained in an MKh-1321A spectrometer with direct introduction of the sample into the ion source, with source temperature 100°C and ionizing electron energy 70 eV.

PMR spectra of 1—2% solutions of the pyrido[1,2-*a*]pyrimidin-4-ones in DMSO-d_6 were registered in a Bruker WH-90 spectrometer with a working frequency of 90 MHz, at 40°C with TMS internal standard. The accuracy of measurement of the spin—spin coupling constants, determined by the digital resolution, was 0.2 Hz. The simulation and iteration of the spectra of compounds I and X were performed in an Aspect-2000 computer by means of the PANIC program.

The UV spectra were taken in a Specord M-40 spectrometer. The ionization constants were measured spectrophotometrically at 25°C. In the measurements of pK_a of protonation, we used hydrochloric acid solutions with a known concentration of hydrogen ions.

Elemental analyses for C, H, and N matched the calculated values.

9-Methoxy-2-methyl-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (Ia, $C_{10}H_{10}N_2O_2$). In 5 ml of DMSO, 0.2 g of solid KOH was dissolved with heating, and 0.176 g (1 mmole) of the 9-hydroxypyridopyrimidinone I was added. After cooling the solution to room temperature, 0.19 ml (3 mmoles) of methyl iodide was added, and the mixture was stirred for 30 h at 40°C.

The reaction mass was diluted with water and extracted with chloroform; the extract was dried over Na_2SO_4 and evaporated to minimum volume; the residue was chromatographed in a column with silica gel, using a 10:1 mixture of chloroform and alcohol as the eluent. Obtained 0.019 g (9%) of compound Ia.

2-Methyl-8-dimethylaminomethyl-9-hydroxy-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (II, $C_{12}H_{15}N_3O_2$). A solution of 0.88 g (0.005 mole) of 3-methyl-9-hydroxypyrido[1,2-*a*]pyrimidin-4-one (I) and 0.67 ml of *N,N,N',N'*-tetramethylmethylenediamine in 5 ml of benzene was refluxed while stirring until the precipitate had completely dissolved; the solvent was driven off, and the resulting precipitate was filtered off and washed with acetone. Obtained 0.4 g (80%) of compound II.

2-Methyl-8-nitro-9-hydroxy-pyrido[1,2-*a*]pyrimidin-4-one (III, $C_9H_7N_3O_4$). To a solution of 0.88 g (0.005 mole) of compound I in 1 ml of concentrated H_2SO_4, with cooling and constant stirring, 0.35 g of HNO_3 (d = 1.5) and 0.5 ml of concentrated H_2SO_4 were added in such a manner that the temperature did not rise above 20°C. The reaction mixture was stirred for 3 h at 20—22°C and then left overnight. The mixture was poured onto 20 g of ice and cautiously neutralized with 25% aqueous ammonia until no more precipitate was formed (pH 3—4); the precipitate was filtered off and air-dried. Yield 0.6 g (54%).

2-Methyl-3-chloro-9-hydroxy-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (IV, $C_9H_7N_2ClO_2$). A suspension of 0.88 g (0.005 mole) of the 9-hydroxypyridopyrimidinone I in 20 ml of concentrated HCl was heated on a steam bath until dissolved; 6 ml of 15% H_2O_2 was added, and the heating was continued for 1 h. After cooling, the precipitate was filtered off and recrystallized from a 7:1 alcohol—water mixture. Yield 0.5 g (48%).

2-Methyl-3,8-dibromo-9-hydroxy-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (V, $C_9H_7N_2Br_2O_2$). To a solution of 0.88 g (0.005 mole) of the pyridopyrimidinone I in 20 ml of acetic acid, a 30% excess of dioxane dibromide was added in portions; the mixture was stirred for 1 h; the precipitate was separated, washed with acetone, dried, and recrystallized from absolute alcohol. Yield 1.0 g (48%).

2-Methyl-8-iodo-9-hydroxy-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (VI, $C_9H_7IN_2O_2$). To a solution of 0.88 g (0.005 mole) of compound I and 0.38 g (0.005 mole) of iodine in 20 ml of alcohol, 0.9 ml of 30% H_2O_2 was added dropwise. After 30 h, the precipitate was filtered off, washed with acetone, and recrystallized from aqueous alcohol. Yield 0.6 g (28%).

2-Methyl-8-phenylazo-9-hydroxy-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (VII, $C_{15}H_{12}N_4O_2$). To a solution of 0.88 g (0.005 mole) of compound I in 20 ml of a 1% potassium hydroxide solution, chilled to 3°C, a solution of 0.47 ml of aniline and 0.36 g (0.005 mole) of sodium nitrite in 1.2 ml of concentrated HCl and a potassium hydroxide solution were added with stirring in such a manner that the pH of the medium was limited to 9. The reaction mass was held for 40 min, after which it was neutralized with gaseous CO_2; the resulting precipitate was separated, washed with water, dried, and crystallized from alcohol. Yield 0.75 g (54%).

2-Methyl-8-amino-9-hydroxy-4*H*-pyrido[1,2-*a*]pyrimidin-4-one (VIII, $C_9H_9N_3O_2$). To a solution of 0.57 g (0.026 mole) of the nitro derivative III in 100 ml of DMFA, 0.6 ml of hydrazine hydrate was added; then, with stirring, 1.5 g of Ni/Re in DMFA was added gradually [6]. The reaction mixture was heated to 50°C over the course of 30 min. The reaction was completed at room temperature, the reaction mixture was filtered, and the solvent was removed by evaporating the filtrate to dryness. The dry residue was recrystallized from water. Obtained 0.35 g (71%) of the amine VIII.

An analogous procedure was used to reduce the azo compound VII. A mixed melting point test on samples of VIII obtained by reduction of the nitro and azo derivatives gave no depression of the melting point, and the PMR spectra of the two samples were identical.

2,9-Dimethyl-7*H*-oxazolo[5,4-3,4]pyrido[1,2-*a*]pyrimidin-4-one (IX, $C_{11}H_9N_3O_2$). A mixture of 0.57 g (0.003 mole) of compound VIII and 1.38 g (0.015 mole) of acetic anhydride was heated for 24 h. The reaction mass was neutralized with an aqueous NaOH solution (with cooling) and extracted with benzene (5 × 30 ml); the extract was washed with aqueous NaCl (10 ml), dried over Na_2SO_4, and evaporated down. Obtained 0.25 g (42%) of compound IX.

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