ACETALS OF LACTAMS AND ACID AMIDES.

XXVII.* NEW SYNTHESIS OF PYRIDO[2,3-d]PYRIMIDINE DERIVATIVES

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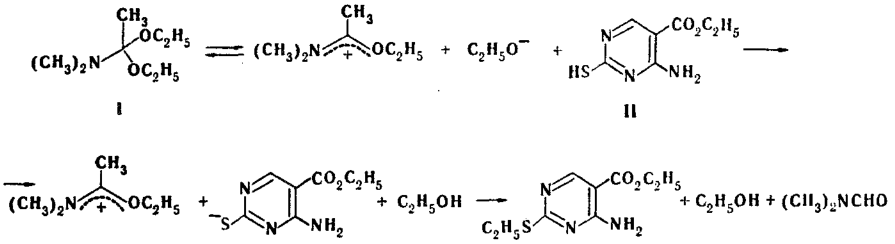
7-Dimethylamino-5-hydroxypyrido[2,3-d]pyrimidine and pyrrolo-, pyrido, and azepino[2,3-b]pyrido[2,3-d]pyrimidine derivatives were synthesized by reaction of acetals of amides and lactams with 4-amino-5-carbethoxypyrimidine derivatives and subsequent intramolecular cyclization of the intermediate amidines. The reactions of these compounds with phosphorus oxychloride and their alkylation reactions were studied.

Pyrido[2,3-d]pyrimidine derivatives are attracting the attention of researchers in connection with their high and diversified biological activity. Substances with pronounced antifolic, antibacterial, diuretic, bacteriostatic, sedative, and coronary-dilating activity have been found among such compounds [2-8]. In particular, the highly effective antibacterial agent pyromidic acid is also a member of this class of compounds [3]. The most important methods for the synthesis of pyrido[2,3-d]pyrimidines are based on the reactions of derivatives of 2-aminonicotinic acid, 2-aminonicotinaldehyde [2, 5, 9-12], and 3-carbethoxy-2-pyridone [13] or its lactim ester [14] with "amidine components" and on the reaction of 6-aminopyrimidine derivatives with β-dicarbonyl compounds [6, 7, 12, 15-17].

We have developed a new method for the synthesis of pyrido[2, 3-d]pyrimidines and their condensed analogs [18, 19] with acetals of amides and lactams and 4-amino-5-carbethoxypyrimidine derivatives as the starting compounds. In the first stage we studied the reaction of dimethylacetamide diethylacetal (I) with 2-mercapto-4-amino-5-carbethoxypyrimidine (II). However, we were unable to isolate the expected amidine — the principal pathway of the process was S-ethylation of the mercaptopyrimidine, evidently via the usual scheme for the alkylation of weak acids with amide acetals [20].



This reaction is of definite interest, since cases in which alkylation of aminomercaptopyrimidines in alkaline media proceeds ambiguously (at the NH_2 and SH groups) are known [21]. The use of amide acetals in such reactions may ensure selective alkylation at the thiol group.

The reaction of acetal I and lactam acetals III-V with 4-amino-5-carbethoxypyrimidines VI and VII give amidines VIII and IX. Attempts to cyclize them under the conditions used to prepare condensed 4-quinolones (200-220°C, TsOH) [22] were unsuccessful because of resinification.

Taking into account the fact that the deuterium exchange of the protons of the α-CH_2 groups of amidines is catalyzed by bases [23] we made an attempt to cyclize amidines VIII and IX under base catalysis conditions. We found that the addition of these amidines to a

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*See [1] for communication XXVI.

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TABLE 1. PMR Spectra of X-XII in d_6-DMSO (ppm)

Compound	SCH_3	NCH_3	2-CH	4-CH	6-CH_2	7-CH_2	8-CH_2	9-CH_2
X*	—	3,00	3,02	8,83	—	—	—	—
XIa	—	3,20	3,09	8,86	2,50	1,88	3,35	—
XIb	2,55	3,20	—	8,91	2,60	1,88	3,42	—
XIIa	—	3,13	3,14	8,87	2,71	1,79		3,46
XIIb	2,56	3,09	—	8,92	2,66	1,79		3,45

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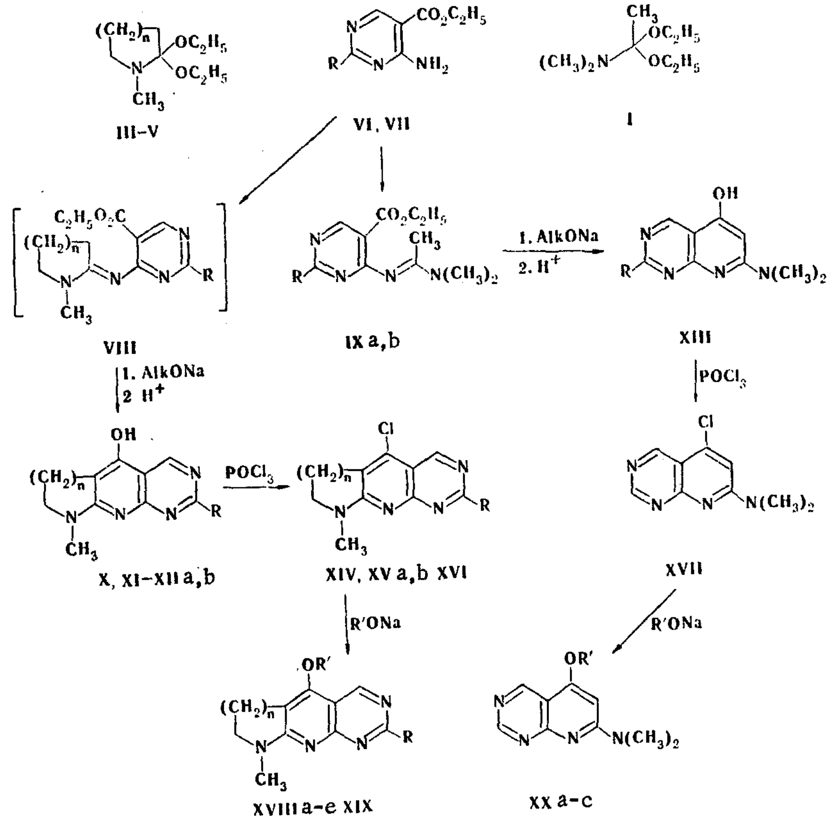
*The signals of the protons of the 6- and 7-CH_2 groups are overlapped by the signals of the solvent.

refluxing solution of sodium alkoxide gives rise to exothermic intramolecular cyclization to give derivatives of pyrido[2,3-d]pyrimidine and pyrrolo-, pyrido-, and azepino[2,3-b]pyrido-[2,3-d]pyrimidines (X-XIII). The structures of the resulting two- and three-ring compounds are confirmed by their PMR spectra (Tables 1 and 2).



III n=1; IV n=2; V n=3; VI R=H; VII R=SCH_3; IX a R=H; b R=SCH_3; X n=1, R=H; XI n=2, a R=H; b R=SCH_3; XII n=3, a R=H; b R=SCH_3; XIII a R=H; b R=SCH_3; XIV n=1, R=H; XV n=2, a R=H; b R=SCH_3; XVI n=3, R=H; XVIII n=2, a R'=C_2H_5; b R'=$(CH_3)_2N(CH_2)_2$; c R'=$(CH_3)_2N(CH_2)_3$; d R'=$C_6H_5CH_2$ (everywhere R=H); e R'=C_2H_5, R=OC_2H_5; XIX n=3, R'=CH_3; R=H; XX a R'=CH_3; b R'=$(CH_3)_2N(CH_2)_3$; c R'=$(CH_3)_2N(CH_2)_3$

The hydroxy group in X-XIII is replaced by chlorine when they are refluxed with phosphorus oxychloride. The halogen is replaced by an alkoxy group when chloro derivatives XIV-XVII are refluxed briefly with solutions of sodium alkoxides. A number of compounds (XVIII-XX), which are analogs of alkoxy-substituted quinolines and condensed pyrrolo-, pyrido-, and azepinoquinolines that have definite biological activity [24, 25], have been obtained by this method. It should be noted that an attempt to carry out similar substitution in the case of three-ring compounds that contain a chloroquinoline fragment was unsuccessful [22]. Not only the halogen but also the methylmercapto group are replaced by ethoxy groups in the reaction of XVb with sodium ethoxide to give diethoxy derivative XVIIIe.

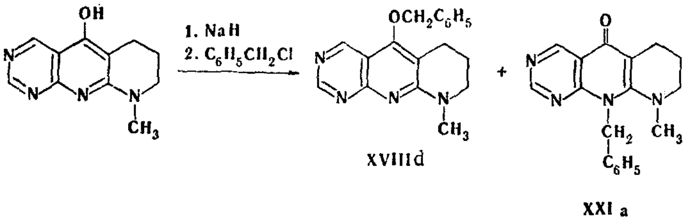
The increase in the lability of the halogen atom on passing from quinoline to pyridopyrimidine derivatives can be explained by a substantial increase in the electron-acceptor properties of the pyrimidine ring as compared with the benzene ring.

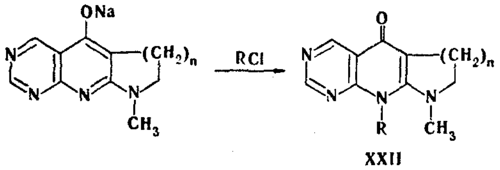
TABLE 2. PMR Spectra of XIII in d_6-DMSO (ppm)

Compound	CH_3	$N(CH_3)_2$	2-CH	4-CH	6-CH
XIIIa	—	3,16	9,10	8,92	6,02
XIIIb	2,54	3,14	—	8,80	5,97

In the benzylation of pyrimidonaphthyridine XIa with benzyl chloride in dimethylformamide (DMF) in the presence of NaH at increased temperatures we isolated two products, one of which proved to be identical to benzyloxy derivative XVIIId, the other of which was found to be the isomeric 10-benzyl derivative (XXIa).

It is interesting to note that only 10-N-benzyl- and 10-N-dimethylaminoethyl derivatives (XXI and XXII) can be isolated in satisfactory yields in the reaction of alkyl halides with the Na salts of pyrimidonaphthyridine XIa and azepinopyridopyrimidine XIIa at room temperature. The alkylation of compounds that contain a γ-pyridone ring with alkyl halides may proceed with the formation of both N-alkyl [26-28] and O-alkyl [29] derivatives.





XXI n=2, a R=$C_6H_5CH_2$; b R=$(CH_3)_2N(CH_2)_2$; XXII n=3, R=$C_6H_5CH_2$

EXPERIMENTAL

The PMR spectra of the compounds were recorded with a JNM-4H-100 spectrometer with tetramethylsilane as the internal standatd. The UV spectra of ~10^{-4} M solutions of the compounds in alcohol were recorded with an EPS-3 spectrophotometer.

2-Ethylmercapto-4-amino-5-carbethoxypyrimidine. A 5-g (31 mmole) sample of acetal I was added to a suspension of 5 g (25 mmole) of pyrimidine II in 35 ml of DMF, and the mixture was heated at 80°C for 2 h. It was then cooled, and the residual (0.3 g) pyrimidine II was removed by filtration, and the DMF was removed by distillation. The residual mass was extracted with boiling hexane to give 3.5 g (65%) of a product with mp 102-103°C [30] (hexane).

N,N-Dimethyl-N'-(2-methylmercapto-5-carbethoxy-4-pyrimidinyl)acetamidine (IXb). A 15-ml sample of anhydrous toluene and 6.1 g (38 mmole) of acetal I were added to 4.2 g (21.1 mmole) of pyrimidine VII, and the mixture was refluxed for 3 h. The toluene was removed by evaporation, and the residue was distilled to give 3 g (51%) of amidine IXb with bp 191-194°C (1 mm) and mp 62-64°C. PMR spectrum, δ: 2.14 (C—CH_3), 2.56 (SCH_3), 3.18 [$(CH_3)_2$], 8.71 (4-CH) and 1.37, 4.31 ppm ($COOC_2H_5$). Found: C 51.0; H 6.5; N 19.8; S 11.3%. $C_{12}H_{18}N_4SO_2$. Calculated: C 51.1; H 6.4; N 19.9; S 11.4%.

N,N-Dimethyl-N'-(5-carbethoxy-4-pyrimidinyl)acetamidine (IXa). This compound, with bp 159-160°C (1 mm), was similarly obtained in 41% yield. PMR spectrum, δ: 2.04 (C—CH_3), 3.10 [$N(CH_3)_2$], 8.64 and 8.72 (2-CH and 6-CH), 1.33 [CH_3(COOEt)], 4.23 ppm [CH_2(COOEt)].

5-Oxo-7-dimethylaminopyrido[2,3-d]pyrimidine (XIIIa). A mixture of 5 g (0.03 mole) of pyrimidine VI, 6.5 g (0.04 mole) of acetal I, and 15 ml of anhydrous toluene was refluxed for 3 h, after which the toluene was removed by evaporation, the residue was added to a refluxing solution of sodium butoxide (from 1.3 g of Na and 40 ml of n-butanol), and the mixture was refluxed for 30 min. The precipitated Na salt of XIIIa was removed by filtration and dissolved in ice water. The solution was acidified to pH 6 with 1 N HCl and worked up to give 2.2 g (42%) of pyridopyrimidine XIIIa with mp 241-243°C (water). Found: C 54.4; H

TABLE 3. 5-Oxopyrido[2,3-d]pyrimidine Derivatives

Compound	mp, °C	Found, %				Empirical formula	Calc., %				Yield, %
		C	H	N	S		C	H	N	S	
X	292[a]	59,6	5,6	27,8	—	$C_{10}H_{10}N_4O$	59,4	5,0	27,7	—	42
XIa	260—263[b]	56,8	5,9	24,2	—	$C_{11}H_{12}N_4O \cdot H_2O$[c]	56,4	6,0	23,9	—	60
XIb	244—248[d]	51,6	5,7	20,2	11,8	$C_{12}H_{14}N_4SO \cdot H_2O$[e]	51,4	5,7	20,0	11,4	50
XIIa	152—154[d]	60,7	6,2	23,8	—	$C_{12}H_{14}N_4O \cdot 0{,}5H_2O$	60,3	6,3	23,4	—	65
XIIb	246—248[d]	56,4	5,8	20,4	11,7	$C_{13}H_{16}N_4SO$	56,5	5,8	20,3	11,6	19

a) From DMF. b) From water. c) Found: H_2O 7.4%. Calculated: H_2O 7.7%. d) From methanol. e) Found: H_2O 6.3%. Calculated: H_2O 6.3%.

5.6; N 28.2%. $C_8H_{10}N_4O \cdot 0.5H_2O$. Calculated: C 54.3; H 5.5; N 28.1%. Compounds X-XII were similarly obtained (Table 3).

2-Methylmercapto-5-oxo-7-dimethylaminopyrido[2,3-d]pyrimidine (XIIIb). A 3.44-g (12.2 mmole) sample of amidine IXb was added to a refluxing solution of sodium butoxide (from 0.56 g of Na and 15 ml of n-butanol), and the mixture was refluxed for 30 min. It was then cooled, and the precipitated Na salt of XIIIb was removed by filtration and dissolved in ice water. The solution was neutralized to pH 7 with 1 N HCl, and the precipitated XIIIb was removed by filtration, washed with a small amount of ice water, and dried to give 1.2 g (42%) of pyridopyrimidine XIIIb with mp 149-150°C (water). Found: C 50.6; H 5.4; N 23.6%. $C_{10}H_{12}N_4SO$. Calculated: C 50.9; H 5.1; N 23.7%.

5-Chloro-9-methyl-6,7,8,9-tetrahydropyrido[2,3-b]pyrido[2,3-d]pyrimidine (XVa). A 5.3-g (24.5 mmole) sample of XIa was refluxed with $POCl_3$ for 2 h, after which the oxychloride was removed by distillation, and the residue was treated with a mixture of 2 N NaOH solution and chloroform. The chloroform was separated, dried with Na_2SO_4, and evaporated to give 5.55 g (97%) of XVa with mp 125-128°C (benzene). UV spectrum, λ_{max} (log ε): 246 (4.20), 285 (3.78), 358 nm (4.02). Found: C 56.1; H 4.9; Cl 14.7; N 23.9%. $C_{11}H_{11}ClN_4$. Calculated: C 56.3; H 4.7; Cl 15.1; N 23.9%.

Compounds XIV, XVI, and XVIII were similarly synthesized (see Table 4).

2-Methylmercapto-5-chloro-9-methyl-6,7,8,9-tetrahydropyrido[2,3-b]pyrido[2,3-d]pyrimidine (XVb). A 2.62-g (0.01 mole) sample of XIb was refluxed with $POCl_3$ for 3 h, after which the oxychloride was removed by distillation, and the residue was dissolved in water. The solution was made alkaline to pH 10, and the resulting precipitate was removed by filtration, washed with water, and dried to give 2.5 g (90%) of chloro derivative XVb with mp 190-192°C (methanol). Found: C 51.4; H 4.5; Cl 12.8; N 19.8; S 11.6%. $C_{12}H_{13}ClN_4S$. Calculated C 51.3; H 4.6; Cl 12.7; N 20.0; S 11.4%.

2,5-Diethoxy-9-methyl-6,7,8,9-tetrahydropyrido[2,3-b]pyrido[2,3-d]pyrimidine (XVIIIe). A 2.1-g (7.5 mmole) sample of XVb was refluxed with a solution of sodium ethoxide (from 2 g of Na and 30 ml of absolute alcohol) for 2 h, after which the alcohol was removed by evaporation, and the residue was treated with water and extracted with chloroform. The extract was dried with Na_2SO_4, and the chloroform was evaporated to give 1.3 g (60%) of XVIIIe with mp 146-148°C (alcohol). Found: C 62.7; H 6.9; N 19.6%. $C_{15}H_{20}N_4O_2$. Calculated: C 62.5; H 6.9; N 19.4%.

Compounds XVIIIa-d, XIX, and XX were similarly synthesized (see Table 4).

5-Benzyloxy-9-methyl-6,7,8,9-tetrahydropyrido[2,3-b]pyrido[2,3-d]pyrimidine (XVIIId) and 5-Oxo-9-methyl-10-benzyl-6,7,8,9-tetrahydropyrido[2,3-b]pyrido[2,3-d]pyrimidine (XXIa). A 0.42-g sample of NaH was added at 75-80°C to 2.5 g (11.5 mmole) of XIa in 40 ml of anhydrous DMF, and the mixture was heated at this temperature for 30 min. A solution of 1.98 g (15.5 mmole) of benzyl chloride in 5 ml of anhydrous DMF was added dropwise, and the mixture was heated at 100°C for 2 h. The DMF was removed by evaporation, water was added to the residue, and the mixture was extracted with chloroform. The extract was dried with Na_2SO_4, the chloroform was evaporated, and the residue was triturated with ethyl acetate to give 0.8 g of XXIa with mp 179-186°C. The ethyl acetate was evaporated, and the residual oil was triturated with acetone to give 0.09 g (2.5%) of XVIIId. The acetone was evaporated, and the residue was treated with a mixture of 2 N aqueous alkali and chloroform. The chloroform extract was

TABLE 4. 5-Chloro and 5-Alkoxy Derivatives of Pyrido[2,3-d]-pyrimidine and Condensed Analogs

Compound	mp, °C	Found, %				Empirical formula	Calc., %				Yield, %
		C	H	Cl	N		C	H	Cl	N	
XIV	189—191[a]	54,1	4,0	16,1	25,2	$C_{10}H_9N_4Cl$	54,4	4,1	16,0	25,4	47
XVI	128—134[b]	57,4	5,4	14,4	22,6	$C_{12}H_{13}N_4Cl$	58,0	5,2	14,3	22,5	93
XVII	177—180[a]	51,9	4,3	17,5	26.8	$C_9H_9N_4Cl$	51,8	4,3	17,0	26,9	86
XVIIIa	127—128[b]	64,2	6,7	—	23.0	$C_{13}H_{16}N_4O$	63,9	6,6	—	23,0	100
XVIIIb	61—64[c]	60,9	7,3	—	23,7	$C_{15}H_{21}N_5O \cdot 0{,}5H_2O$	60.8	7,4	—	23.7	65
XVIIIb · HCl	188—189[a]	—	—	17,8	18,0	$C_{15}H_{21}N_5O \cdot 2HCl \cdot$ $\cdot C_2H_5OH$	—	—	17.6	17,3	
XVIIIc	85—87[d]	63.5	7.5	—	23,5	$C_{16}H_{23}N_5O$	63,8	7,7	—	23,2	92
XVIIId	148—149[a]	70,3	6,1	—	18,5	$C_{18}H_{18}N_4O$	70,6	5,9	—	18,3	33
XIX	115—117[d]	64,0	6,6	—	23,1	$C_{13}H_{16}N_4O$	63,9	6,6	—	23,0	90
XIX	180—182[e]	58,8	5,8	—	27,3	$C_{10}H_{12}N_4O$	58,8	5,9	—	27,4	90
XXb	135—138[e]	59,3	7,4	—	26.6	$C_{13}H_{19}N_5O$	59,8	7,3	—	26,8	93
XXc	108—111[d]	60,8	8,0	—	25,5	$C_{14}H_{21}N_5O$	61,1	7,6	—	25,5	76
XXc · HCl	218—220[a]	46,0	6,7	19,4	18,8	$C_{14}H_{21}N_5O \cdot 2HCl \cdot H_2O$	46,0	6,9	19,4	19,1	

a) From alcohol. b) From benzene. c) From hexane. d) From heptane. e) From ethyl acetate.

dried with Na_2SO_4, the chloroform was evaporated, the residue was dissolved in acetone, and water was added (the acetone–water ratio was 1:1). The suspension of solid reaction product in aqueous acetone was decanted from the oil and filtered to give 0.75 g of XXIa. The overall yield of XXIa, with mp 204-207°C (from acetone), was 1.55 g (44%).

5-Oxo-9-methyl-10-benzyl-6,7,8,9-tetrahydropyrido[2,3-b]pyrido[2,3-d]pyrimidine (XXIa). A solution of 1.3 g of benzyl chloride in 10 ml of anhydrous DMF was added dropwise at room temperature to 2.4 g of the Na salt of XIa in 20 ml of anhydrous DMF, and the mixture was allowed to stand for 12 h. It was then filtered, and the DMF was removed by evaporation. The residue was triturated with water to give 2 g (65.5%) of pyrimidonaphthyridine XXIa with mp 204-207°C (acetone). UV spectrum, λ_{max} (log ε): 242 (4.36) 256 (3.95); 364 nm (4.01). PMR spectrum, δ: 3.12 (NCH_3), 5.57 (NCH_2), 8.78 (4-CH), 3.20 (2-CH), 1.76 (7-CH_2), 2.48 (6-CH_2), 3.30 (8-CH_2) and 7.30 ppm (C_6H_5). Found: C 67.2; H 5.7; N 17.3; H_2O 5.6%. $C_{18}H_{12}N_4O \cdot H_2O$. Calculated: C 66.8; H 6.2; N 17.3; H_2O 5.6%.

The addition of an alcohol solution of oxalic acid to a solution of XXIa in absolute alcohol gave oxalate XXIa with mp 225°C (dec., methanol). Found: C 60.5; H 5.3; N 14.3%. $C_{20}H_{20}N_4O_5$. Calculated: C 60.6; H 5.1; N 14.1%.

5-Oxo-9-methyl -(10-β-N,N-dimethylamino)ethyl-6,7,8,9-tetrahydropyrido[2,3-b]pyrido[2,3-d]-pyrimidine (XXIb). The procedure used to prepare XXIa was also employed in this case, except that XXIb was isolated in the form of the oxalate, with mp 207-210°C (methanol), in 46% yield. Base XXIb, with mp 179-185°C (ethyl acetate), was obtained from the oxalate. UV spectrum, λ_{max} (log ε): 240 (4.37); 275 (3.37); 362 nm (4.03). PMR spectrum, δ: 3.26 (NCH_3), 5.10 (NCH_2), 8.92 (2-CH), 8.88 (4-CH), 2.80 (6-CH_2), 1.88 (7-CH_2), 3.50 (8-CH_2) and 7.40 ppm (C_6H_5). Found: C 62.9; H 7.4; N 24.6%. $C_{15}H_{21}N_5O$. Calculated: C 62.7; H 7.3; N 24.4%.

5-Oxo-10-methyl-11-benzyl-6,7,8,9-tetrahydro-10H-azepino[2,3-b]pyrido[2,3-d]pyrimidine (XXII). A solution of 0.85 g of benzyl chloride in 5 ml of anhydrous DMF was added to 1.6 g (6.35 mmole) of the Na salt of XIIIa in 10 ml of anhydrous DMF, and the mixture was heated at 100°C for 2 h. The DMF was then removed by evaporation, and the residue was treated with water and extracted with chloroform. The extract was dried with Na_2SO_4, the chloroform was evaporated, and the residue was triturated with ether to give 0.8 g (39%) of XXII with mp 194-196°C (benzene). UV spectrum, λ_{max} (log ε): 250 (4.31), 280 (3.94), 370 nm (3.58). Found: C 67.6; H 6.7; N 16.6; H_2O 5.0%. $C_{19}H_{20}N_4O \cdot H_2O$. Calculated: C 67.5; H 6.5; N 16.6; H_2O 5.3%.

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