PYRIMIDINES.

70.* RELATIVE REACTIVITIES OF THE CHLORINE ATOMS OF 2,2',4-TRICHLORO-4',5-DIPYRIMIDINYL IN ITS REACTION WITH PIPERIDINE

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UDC 547.853.7.855.1'542.953.5

2,2',4-Trichloro-6'-phenyl-4',5-dipyrimidinyl, for which nucleophilic substitution with piperdine under various conditions was studied, was obtained from 2,2',4-trioxo-6'-phenyl-1,1',2,2',3,4-hexahydro-4',5-dipyrimidinyl. It is shown that there is an appreciable difference in the rates of substitution of the first, second, and third chlorine atoms, and this made it possible to obtain reaction products that contain one, two, and three piperidino groups. The chlorine atom in the 4 position is replaced initially, after which the chlorine atom in the 2 position undergoes substitution. The structures of the compounds were proved by chemical transformations and analysis of the PMR spectra.

Research involving the study of the chemical properties of symmetrical 5,5'-dipyrimidinyls has been carried out [2, 3]. It is evident that in the case of unsymmetrical dipyrimidinyls a number of peculiarities in the behavior of these systems that are associated with the dissimilar effects of the rings on one another may be manifested. In the case of 4',5-dipyrimidinyl I we investigated some reactions that are typical for pyrimidine systems. We obtained trichloro derivative II from 2,2',4-trioxo-6'-phenyl-1,1',2,2',3,4-hexahydro-4',5-dipyrimidinyl [1] and investigated the possibility of stepwise (selective) nucleophilic substitution of the chlorine in II.

When oxopyrimidine I is refluxed with $POCl_3$ in the presence of a dialkylaniline [4], it is converted to a trichlorodipyrimidinyl (II); however, the yields are low and difficult to reproduce. When dimethylaniline was used, aminodichlorodipyrimidinyls IIIa, b were always present among the reaction products. The difficulties involved in the preparation of trichlorodipyrimidinyl II are probably associated with the possibility of the existence of an intramolecular hydrogen bond [5] in the starting trioxodipyrimidinyl [1]. It has been shown that cleavage of the hydrogen bonds by protonation of the molecule makes it possible to obtain chloro derivatives without any difficulty [5, 6]. In fact, when water was added [5] to the reaction mixture, trichlorodipyrimidinyl II was obtained in good yield.

The nucleophilic substitution of chlorine in dipyrimidinyl II by means of piperidine was studied. On the basis of the numerous literature data on the nucleophilic substitution of 2- and 4-halopyrimidines in solvents such as alcohols, amines, and dimethyl sulfoxide (DMSO) [7], one might have expected that in the case of trichlorodipyrimidinyl II initial attack by the nucleophile should occur at the 4 position of the A ring when the reaction is carried out in methanol (see the scheme given on the next page).† However, the carrying out of the same reaction in isooctane could change the relative order of replacement of the chlorine atoms [8] and lead to the formation of isomeric 2- or 4-piperidino derivatives.

We carried out the monoamination of trichloro derivative II with piperidine in methanol and isooctane and, according to the data from thin-layer chromatography (TLC) and the IR spectra, obtained the same product (IV) in both cases. However, the conformity in these data

*See [1] for communication 69.

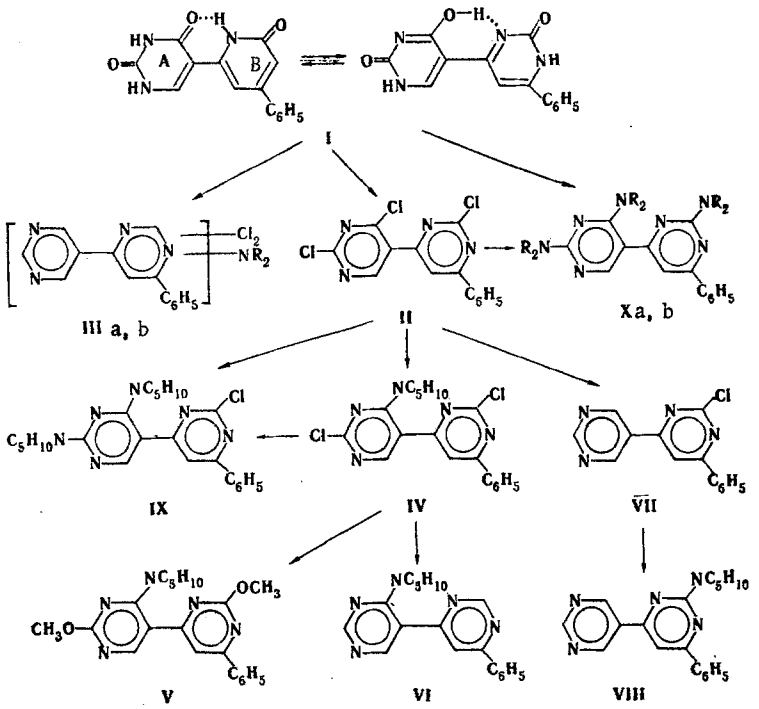
†Similar designations of the pyrimidine rings are subsequently used for all of the compounds.

Novosibirsk Institute of Organic Chemistry, Siberian Branch of the Academy of Sciences of the USSR, Novosibirsk 630090. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 6, pp. 821-826, June, 1979. Original article submitted August 7, 1978.

0009-3122/79/1506-0671$07.50 © 1979 Plenum Publishing Corporation

did not exclude the possibility of the formation of mixtures of chromatographically inseparable isomeric dichloropiperidino derivatives. This sort of situation has been described in the case of the monoamination of dichloropyrimidines, in which case the mixture of isomers could be separated only after their conversion to the corresponding methoxy amino [9] or thio amino derivatives [10].

To verify the individuality of the monosubstitution product we treated crude product IV with sodium methoxide. The dimethoxypiperidino derivative (V) obtained gave one spot on the chromatogram (in systems a, b, and c), and its PMR spectrum contained two signals of CH_3 groups. In the case of the existence of a mixture of isomers one might have expected a large number of signals, since, e.g., the signals of the 2-OCH_3 and 4-OCH_3 groups in the PMR spectrum of 2,4-dimethoxypyrimidine differ [11]. The data that we obtained indicated that both dipyrimidinyl V and starting IV are individual substances and contain virtually no admixed second isomer.



III a, b; II; Xa, b; IX; IV; VII; V; VI; VIII

III a $R=CH_3$; b $R_2=CH_3, C_6H_5$; X a $R_2=(CH_2)_5$; b $R=CH_3$

To ascertain the position of the piperidino group in dipyrimidinyl IV we used the method of catalytic dechlorination with subsequent analysis of the PMR spectra of the products of the reduction of dipyrimidinyl IV and trichloro derivative II and comparison with the PMR spectrum of 6'-methyl-4',5-dipyrimidinyl [12].

The PMR spectrum of VI obtained by reduction of IV contains two doublets at 7.79 and 9.19 ppm with a splitting constant of 2 Hz; these signals can be assigned only to 5'-H and 2'-H of the B ring, in agreement with the data in [12] and the assumption that primarily the chlorine atom in the A ring undergoes substitution. The PMR spectrum of product VI contains two other signals (see Table 1), the presence of which indicates the existence of an unsymmetrical structure for the 4-substituted ring, since the signals of the 4-H and 6-H protons should not differ [12]. Thus VI has the 4-piperidyl-6'-phenyl-4',5-dipyrimidinyl structure, i.e., the chlorine atom in the 4 position primarily undergoes substitution in the case of nucleophilic substitution in trichlorodipyrimidinyl II.

The reduction of trichlorodipyrimidinyl II under the same conditions did not lead to the expected product of complete reduction, viz., 6'-phenyl-4',5-dipyrimidinyl. Chlorodipyrimidinyl VII was unexpectedly obtained as the principal product. Since the integral intensity of the signal at 9.42 ppm in the PMR spectrum of VII corresponds to two protons, it can be assigned only to 4-H and 6-H of the symmetrical A ring, and the remaining chlorine atom may be in either the 2 or 2' position of the dipyrimidinyl; the signal at 9.35 ppm will correspondingly be related to either the 2'-H or 2-H proton. The absence of splitting of the 2'-H proton by 5'-H, which was observed in [12] for VI, increases the probability that the chlorine atom is in the 2' position.

TABLE 1. Chemical Shifts in the PMR Spectra of 4',5-Dipyrimidinyl Derivatives (δ, ppm)

Compound	Solvent	2-R	4-R	2'-R	6-H	5'-H	6'-C_6H_5	Intensity ratio
II	$CDCl_3$	—	—	—	9,03	8,13	7,40—7,75; 7,97—8,30	1:1:3:2
IIIa	d_7-DMF		2,93[a]		8,37	7,90?	7,16—7,58 8,06—8,30	6:1:3:3 (?)
IIIb	CCl_4		3,59[a]		8,44	7,24	6,97—7,50; 7,81—8,13	3:1:1:6:4[b]
IV	$CDCl_3$	—	1,57[c] 3,40[d]	—	8,40	7,73	7,33—7,73; 7,90—8,27	6:4:1:1:3:2
V	The same	3,97 (or 4,10)	1,56[c] 3,45[d]	4,10 (or 3,97)	8,56	8,08	7,43—7,75; 8,18—8,46	3:6:4:3:1:1:3:2
VI	„ „	8,43 Broad	1,50[c] 3,30[d]	9,19[e]	8,56	7,79[e]	7,29—7,53; 7,90—8,09	1:6:4:1:1:1:3:2
VII	„ „	9,35	9,42[f]	—	[f]	8,00	7,35—7,68; 7,97—8,23	1:2:1:3:2
VIII	„ „	9,26	9,32[f]	1,65[c] 3,90[d]	[f]	7,22	7,30—7,61; 8,26—8,76	1:2:6:4:1:3:2
IX	„ „		1,60[c] 3,33[d] 3,82[d]	—	8,64	7,92	7,43—7,67; 8,00—8,30	12:4:4:1:1:3:2
Xa	„ „		1,58[c], 3,29[d], 3,84[d]		8,53	7,14	7,38—7,65; 8,05—8,28	18:4:8:1:1:3:2
Xb	„ „		2,86; 3,10; 3,27		8,30	6,90	7,23—7,50; 8,02—8,31	6:6:6:1:1:3:2

[a]The position of the substituent was not established. [b]Together with the signals of the N—C_6H_5 group. [c]β- and γ-H-Piperidino groups. [d]α-H-Piperidino groups. [e]Doublet, $J_{2'-5'}$ = 2 Hz. [f]Displayed as one signal.

To definitively ascertain the position of the chlorine atom we used data on the shift of the signals of the protons of the pyrimidine ring when an amino group is incorporated in it [13, 14]. In the case of amination of monochloro derivative VII one should observe a shift of the signals of the protons of only one ring in the PMR spectrum, and the position of the signals of the other should remain virtually unchanged. The 5'-H signal in the PMR spectrum of dipyrimidinyl VIII was shifted 52 Hz to strong field (as compared with VII), whereas the position of the 4-H and 6-H signals changed only slightly; this constitutes unambiguous evidence [13] in favor of the 2'-chlorodipyrimidinyl structure for VII.

The subsequent replacement of chlorine atoms in dipyrimidinyl IV could have taken place at both the 2 and 2' positions. Considering the strong deactivating effect of the NR_2 group on the lability of the chlorine atom [8], one might have expected replacement of the 2'-Cl atom. However, the PMR spectrum of the dipiperidino derivative (IX) obtained contradicted this assumption. It is apparent from the data presented in Table 1 that the position of the signal of the 5'-H proton is similar to the position of the analogous signals for 2'-chloro derivatives II, IV, and VII and differs substantially from the position of the 5'-H signal in substituted 2'-NR_2 dipyrimidinyls VIII and X. At the same time, the signal of the 6-H proton in the spectrum of IX was shifted 24 Hz to strong field [14] as compared with the 6-H signal in the spectrum of starting trichlorodipyrimidinyl II. These data show that replacement of the second chlorine atom also occurs in the A ring.

This result can be explained from the point of view of the mutual effect of the A and B fragments of the dipyrimidinyl. The chlorine atom in the 2 position is deactivated by the 4-NR_2 group but is activated from the 5 position by the 4'-pyrimidinyl acceptor substituent [15], while the reactivity of the 2'-halo atom decreases both under the influence of the 6'-phenyl group [16] and, evidently, under the influence of the 5-pyrimidinyl substituent in the 4' position, which contains, moreover, a strong donor group. However, there are no data available regarding the effect of a heteroaryl group from the 5 position of pyrimidine on the rate of nucleophilic substitution of chlorine in the 2 position [17].

Chlorodipiperidinodipyrimidinyl IX was obtained both by amination of trichlorodipyrimidinyl II in methanol and from IV in isooctane. The reaction in methanol proceeds ambiguously [according to the data from thin-layer chromatography (TLC)], and the yield of IX is lower than in isooctane; however, only an insignificant amount of tripiperidino derivative Xa is present in the reaction products.

Complete replacement of the chlorine atoms by amino groups occurred when II was refluxed with an excess of the corresponding amine in alcohol, and 2,2',4-tripiperidyl- (Xa) and 2,2',4-tris(dimethylamino)-6'-phenyl-4',5-dipyrimidinyl (Xb) were obtained. Compound Xb was also obtained by reaction of trioxodipyrimidinyl I with hexametapol by direct amination [18].

The data obtained in this research make it possible to conclude that there is an appreciable difference in the rates of reactions involving nucleophilic substitution of the chlorine atoms in trichlorodipyrimidinyl II. Whereas replacement of the first chlorine atom is complete in a few minutes in both isooctane and methanol, monopiperidino derivative IV was present in the mixture (according to TLC data) even after 15 h in the case of replacement of the second chlorine atom in isooctane. The development of only traces of trisubstituted derivative Xa in the preparation of dipiperidinodipyrimidinyl IX is an indirect indication of an appreciable difference in the rates of substitution of the 2- and 2'-chloro atoms. This leads to the fact that the reaction is strictly a stepwise process.

EXPERIMENTAL

The UV spectra of solutions of the compounds in alcohol were recorded with a Specord UV-vis spectrophotometer. The PMR spectra were recorded with Varian A56/60A and Brucker WP-80 spectrometers with hexamethyldisiloxane as the internal standard. The molecular weights were determined with an MS-902 high-resolution mass spectrometer with a system for direct introduction of the samples at 110-140°C. Thin-layer chromatography (TLC) was carried out on Silufol UV-254 and KSK silica gel plates in chloroform—alcohol (30:1) (system a) (10:1) (system b) and ethyl acetate (system c) and on activity II Al_2O_3 (elution with benzene).

2,2',4-Trichloro-6'-phenyl-4',5-dipyrimidinyl (II). A) A few drops of water were added carefully to a mixture of 10 g (0.034 mole) of I, 100 ml of $POCl_3$, and 10 ml of dimethylaniline, and the mixture was refluxed for 6 h. The bulk of the $POCl_3$ was removed by vacuum distillation, and the residue was dissolved in 80 ml of benzene. The benzene solution was poured over ice, the benzene layer was separated, and the aqueous layer was extracted with benzene (four 80 ml-portions). The combined benzene solutions were washed with 10% $NaHCO_3$ and water, dried with $CaCl_2$, and evaporated. The residue was washed with ether and recrystallized from n-octane to give 8.5 g (74%) of lemon-yellow II with mp 150-152°C. An analytical sample (which was white) was obtained by sublimation at 170°C (2.5 mm) and had mp 153-154°C and R_f 0.50 (Al_2O_3). UV spectrum, λ_{max} (log ε): 210 (4.40), 252 (4.30), 303 nm (4.26). Found: C 49.9; H 2.1; Cl 31.2; N 16.4%; M 336. $C_{14}H_7Cl_3N_4$. Caluclated: C 49.9; H 2.1; Cl 31.5; N 16.6%; M 336.

B) A mixture of 2 g (7.1 mmole) of dipyrimidinyl I, 3 ml of dimethylaniline, and 26 ml of $POCl_3$ was refluxed for 5 h, after which the $POCl_3$ was removed by vacuum distillation. Benzene (10 ml) and 1 g of K_2CO_3 were added to the residue, and the mixture was cooled and neutralized with 30% K_2CO_3 solution. The mixture was extracted with benzene, and the extract was dried with $MgSO_4$ and concentrated to a volume of 20 ml. In various experiments we were able to isolate up to 10% dipyrimidinyl IIIa, 37% dipyrimidinyl IIIb, and 20% trichloro derivative II from this benzene solution by combined chromatography on plates and with a column (silica gel, Al_2O_3, benzene). Dimethylaminodichlordipyrimidinyl IIIa was obtained as light-green plates that were only slightly soluble in chloroform, alcohol, and dimethyl sulfoxide (DMSO) and had mp 236-237°C (sublimation, from alcohol) and R_f 0.15 (Al_2O_3). Found: M 345.0532. The empirical formula calculated from this molecular weight is $C_{16}H_{13}Cl_2N_5$.

Methylphenylaminodichlorodipyrimidinyl IIIb had mp 161-163°C (from n-octane) and R_f 0.52 (Al_2O_3). Found: N 17.0%; M 407.0717. $C_{21}H_{15}Cl_2N_5$. Calculated: N 17.1%; M 407.0706.

2,2'-Dichloro-4-(N-piperidyl)-6'-phenyl-4',5-dipyrimidinyl (IV). A) A solution of 0.6 ml (6 mmole) of dry piperidine in 10 ml of isooctane was added at 60°C to a solution of 0.68 g (2 mmole) of trichloro derivative II in 360 ml of dry isooctane, and the mixture was maintained at this temperature for 25 min. It was then cooled to room temperature and stored in a refrigerator overnight, and the resulting white precipitate was removed by filtration, washed with water, and dried to give 0.64 g (83%) of dipyrimidinyl IV with mp 195-195.5°C (from alcohol) and R_f 0.34 (system a). UV spectrum, λ_{max} (log ε): 245 (4.53), 286 (4.57), 346 nm (3.92). Found: C 58.9; H 4.6; N 18.2%; M 385. $C_{19}H_{17}Cl_2N_5$. Calculated: C 59.1; H 4.5; N 18.2%; M 385.

B) Monoamino derivative IV was also obtained at room temperature from 0.056 g (0.167 mmole) of trichloro derivative II and 0.033 ml (0.33 mmole) of dry piperidine in 20 ml of isooctane; the yield was 0.048 g (75%).

C) A mixture of 0.056 g (0.167 mmole) of II and 0.033 ml (0.33 mmole) of dry piperidine was stirred in 30 ml of methanol at room temperature for 30 min, during which the mixture turned yellow. It was then evaporated, and the residue was treated with water and extracted with chloroform (five 5-ml portions). The extract was dried with $MgSO_4$, the solvent was removed by evaporation, and the residue was recrystallized from alcohol to give 0.034 g (53%) of pure monopiperidino derivative IV.

4-(N-Piperidyl)-2,2'-dimethoxy-6'-phenyl-4',5-dipyrimidinyl (V). A 0.1-g (0.26 mmole) sample of IV was added to a solution of sodium methoxide, prepared from 0.1 g (4.35 mmole) of sodium and 5 ml of methanol, and the mixture was refluxed for 30 min. It was then cooled to room temperature, and the solution was evaporated. The precipitate was washed with water and dried to give 0.097 g (99%) of dipyrimidinyl V with mp 148-149°C (from methanol) and R_f 0.26 (system a), 0.53 (system b), and 0.77 (system c). UV spectrum, λ_{max} (log ε): 208 (4.65) 235 (4.37), 260 (4.19), 288 (4.28), 335 nm (4.15). Found: N 18.5%; M 377. $C_{21}H_{23}N_5O_2$. Calculated: N 18.6%; M 377.

4-(N-Piperidyl)-6'-phenyl-4',5-dipyrimidinyl (VI). A 0.5-g (1.3 mmole) sample of dichloropiperidino derivative IV was dissolved in 80 ml of alcohol and hydrogenated in the presence of 0.35 g of 10% Pd/C and 0.35 g of MgO at 60°C. After the calculated amount of hydrogen had been absorbed (6 h), the solution was separated from the precipitate, and the precipitate was washed with hot alcohol. The combined solutions were evaporated, and the residual oil was crystallized by the addition of ether to give 0.37 g (90%) of dipyrimidinyl VI with mp 104-106°C (from alcohol) and R_f 0.75 (system b). UV spectrum, λ_{max} (log ε): 207 (4.41), 233 (4.31), 277 (4.39), 385 nm (3.82). Found: M 317.1634. The empirical formula calculated from this molecular weight is $C_{19}H_{19}N_5$.

2'-Chloro-6'-phenyl-4',5-dipyrimidinyl (VII). A 0.1-g (0.3 mmole) sample of trichlorodipyrimidinyl II was dissolved in 35 ml of alcohol and hydrogenated at room temperature in the presence of 0.05 g of 10% Pd/C and 0.15 g of MgO for 2 days, during which period the amount of hydrogen theoretically necessary for complete reduction was absorbed. The solution was separated from the precipitate, and the precipitate was washed with hot alcohol. The combined solutions were evaporated, and the residue was applied to a column (5 by 2 cm) filled with silica gel and chromatographed in system b. Evaporation of the eluate and trituration of the resulting oil with absolute ether gave 0.036 g (45%) of chlorodipyrimidinyl VII. To obtain an analytical sample the product was additionally sublimed at 180° (1.5 mm); the product then had mp 203-204°C and R_f 0.35 (system a). Found: N 20.5%; M 268. $C_{14}H_9ClN_4$. Calculated: N 20.8%; M 268.

2'-(N-Piperidyl)-6'-phenyl-4',5-dipyrimidinyl (VIII). A mixture of 0.1 g (0.37 mmole) of VII and 0.15 ml (1.5 mmole) of dry piperidine in 3 ml of absolute alcohol was refluxed for 5 h, after which it was cooled, and the resulting precipitate was removed by filtration, washed with water, and dried to give 0.097 g (82%) of a slightly colored product. Recrystallization from alcohol gave 0.081 g (70%) of VIII with mp 212-213°C (sublimation) and R_f 0.88 (system b). Found: M 317.1634. The empirical formula calculated from this molecular weight is $C_{19}H_{19}N_5$.

2'-Chloro-2,4'di(N-piperidyl)-6'-phenyl-4',5-dipyrimidinyl (IX). A) A mixture of 0.056 g (0.167 mmole) of II and 0.066 ml (0.66 mmole) of dry piperidine was stirred in absolute isooctane at 60°C for 15 h, after which the mixture was cooled and evaporated, and the residue was separated by TLC on silica gel in system a. Workup of the zone with R_f ~0.3 gave 0.065 g (90%) of IX with mp 189-191°C (from alcohol) and R_f 0.30 (system a). Found: N 19.5%; M 434. $C_{24}H_{27}N_6$. Calculated: N 19.3%; M 434.

B) A mixture of 0.064 g (0.167 mmole) of IV and 0.033 ml (0.33 mmole) of dry piperidine was refluxed in 50 ml of absolute methanol for 10 h. The mixture turned yellow at the end of the reaction. It was worked up as in method A to give 0.05 g (69%) of IX with mp 187-191°C.

2,2',4-Tri(N-piperidyl)-6'-phenyl-4',5-dipyrimidinyl (Xa). A mixture of 0.168 g (0.5 mmole) of trichloro derivative II, 0.60 ml (6 mmole) of dry piperidine, and 3 ml of absolute alcohol was refluxed for 10 h, during which the course of the reaction was monitored by chromatography (system a). The mixture was then stored in a refrigerator for 24 h, and the resulting precipitate was removed by filtration, washed with water, and dried to give 0.237 g (98%) of Xa with mp 159-160°C (from alcohol) and R_f 0.17 (system a). UV spectrum, λ_{max} (log ε) 204 (4.41), 262 (4.69), 322 nm (4.12). Found: C 71.8; H 8.0; N 20.3%; M 483. $C_{29}H_{37}N_7$. Calculated: C 72.0; H 7.7; N 20.3%; M 483.

2,2',4-Tris(dimethylamino)-6'-phenyl-4',5-dipyrimidinyl (Xb). A) A 0.2-g (0.6 mmole) sample of trichloro derivative II was added to a solution of 0.35 g (8 mmole) of dry dimethylamine in 20 ml of absolute alcohol, and the mixture was refluxed for 6 h. The alcohol was then removed by vacuum distillation, and the residue was treated with water and extracted with chloroform (four 20-ml portions). The chloroform solution was washed with water, dried with $MgSO_4$, and evaporated. The resulting oil was dissolved in 3 ml of 50% CH_3COOH, the solution was filtered, and the filtrate was neutralized (with adequate cooling) with 4 N NaOH. The white precipitate was removed by filtration and dried to give 0.13 g (60%) of Xb with mp 109-110°C (from methanol) and R_f 0.20 (system a). UV spectrum, λ_{max} (log ε): 259 (4.62), 323 (4.04), 360 nm (4.12). Found: C 66.5; H 7.0; N 26.6%; M 363. $C_{20}H_{25}N_7$. Calculated: C 66.1; H 6.9; N 26.9%; M 363.

B) A mixture of 2.82 g (0.01 mole) of trioxo derivative I, 5.3 ml (0.03 mole) of hexamethylphosphoric triamide, and 0.35 g (0.004 mole) of dimethylamine hydrochloride was heated at 230-240°C for 1 h, after which it was cooled to room temperature, and the fused mass was triturated with 3% NaOH solution. The solid material was removed by filtration and washed with water to give 3.05 g of a brown product that was subsequently purified with a column filled with silica gel (system a). Evaporation of the fraction with R_f 0.20 gave a colorless oil that began to crystallize when absolute methanol was added. The yield of tris(dimethylamino) derivative Xb, with mp 106-110°C, was 1.93 g (53%).

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