5-Amino-6-nitro-2,4-diethoxypyrimidine (IIb). This compound had mp 128-130°C. UV spectrum (in methanol), λ_{max} (log ε): 415 nm (3.62). PMR spectrum ($CDCl_3$), δ: 1.36 (3H, t, CH_3), 1.42 (3H, t, CH_3), 4.34 (2H, q, CH_2). 4.53 (2H, q, CH_2), 5.88 ppm (2H, broad s, NH_2).

The structure of IIa was also proved by a chemical method. The previously described [3] 5-amino-2,4,6-trimethoxypyrimidine (III) was obtained when it was refluxed with an equimolar amount of sodium methoxide as a result of nucleophilic substitution of the nitro group; the product had mp 104-105°C and was obtained in 73% yield. UV spectrum (in methanol), λ_{max} (log ε): 2.79 nm (3.93). PMR spectrum ($CDCl_3$), δ: 3.03 (2H, broad s, NH_2), 3.79 (3H, s, OCH_3), 3.86 ppm (6H, s, OCH_3).

The molecular masses of 6-nitropyrimidines IIa, b determined by mass spectrometry and the results of elementary analysis of the compounds obtained were in agreement with the calculated values.

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FORMATION OF A CONDENSATION SIDE PRODUCT IN THE SYNTHESIS OF 2-BENZYL-4-METHYL-6-CHLOROPYRIMIDINE

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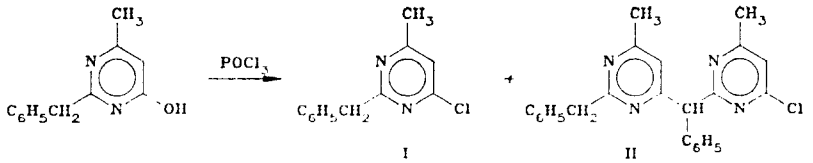
UDC 547.853'855

A general method for obtaining chloropyrimidines is refluxing hydroxypyrimidines in excess phosphorus oxychloride, in individual cases in the presence of N,N-dialkylanilines [1].

In the synthesis of 2-benzyl-4-methyl-6-chloropyrimidine by refluxing the corresponding hydroxypyrimidine with an eightfold excess of phosphorus oxychloride, in addition to the formation of chloropyrimidine I (27%), we obtained, in 44% yield, **2-benzyl-4-methyl-6-[α-4-methyl(6-chloro-2-pyrimidinyl)benzyl]pyrimidine (II)** with mp 195-197°C and R_f 0.54 [Silufol UV 254, benzene–acetone (6:1)]. Mass spectrum, m/z (relative intensity, %): 402 (6), 400 (17), 220 (15), 219 (36), 218 (47), 217 (100), 183 (37), 182 (27), 117 (26), 91 (49), 90 (17).



We do not exclude the possibility that Wintersteiger and coworkers [2] and Ochiai and Janai [3] in their studies of the synthesis of 2-benzyl-4-methyl-6-chloropyrimidine also observed the side process, as evidenced indirectly by the fact that, despite the low yields (50-56%), they avoided prolonged refluxing (no more than 5-7 min). However, a condensation product was not isolated, and the transformed that we noted has not been previously described.

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5,5-DIMETHYL-2-THIOXO-3-PHENYLSPIRO(1,3-OXAZOLIDINE-4,2-[2H]CHROMENES)

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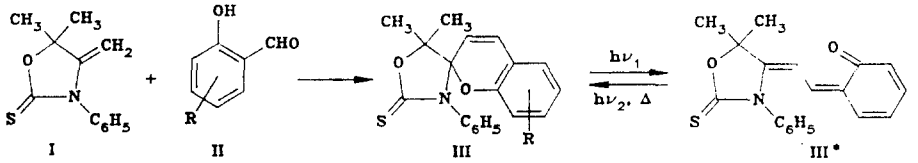
Proceeding from a stable methylene base – 2-thioxo-4-methylene-5,5-dimethyloxazolidine (I) [1] – via the scheme presented below we obtained new photochromic spiropyrans III in 70-90% yields:



IIIa R=H; b R=5′,6′-benzo; c R=6′-NO_2

The condensation was carried out by refluxing oxazolidinethione I and the corresponding o-hydroxy aldehydes II in a molar ratio of 1:1 in glacial acetic acid in the presence of catalytic amounts of $HClO_4$. The spirocyclic structure of the III obtained was confirmed by IR, UV, and PMR spectroscopic data.

The results of elementary analysis of IIIa-c were in agreement with the calculated values.

Compound IIIa. This compound was obtained in 71% yield and had mp 153-154°C (from ethanol). IR spectrum (mineral oil): 1746, 1646, 1600, 1587, 1309, 1256, 1237, 1157, 1100, 1029, 1009, 985, 960 cm^{-1}. PMR spectrum ($CDCl_3$): 1.48 [6H, s, $C(CH_3)_2$], 5.42 (1H, d, $^3J_{CH=CH}$ = 10 Hz, 3'-H), 6.55 (1H, d, $^3J_{CH=CH}$ =10 Hz, 4'-H), 6.75-7.38 ppm (9H, m, aromatic).

Compound IIIb. This compound was obtained in 86% yield and had mp 193-194°C (from ethanol). IR spectrum (mineral oil): 1730, 1660, 1633, 1580, 1302, 1266, 1237, 1100, 1075, 1036, 1009, 980, 960 cm^{-1}. PMR spectrum ($CDCl_3$): 1.55 [6H, s, $C(CH_3)_2$], 5.97 (1H, $^3J_{CH=CH}$ = 10.5 Hz, 3'-H), 7.05-7.93 ppm (12H, m ,4'-H, aromatic).

Compound IIIc. This compound was obtained in 81% yield and had mp 176-177°C (from ethanol). IR spectrum (mineral oil): 1761, 1646, 1612, 1573, 1304, 1256, 1232, 1107, 1100, 1075, 1011, 980, 951 cm^{-1}. PMR spectrum ($CDCl_3$): 1.53 [6H, s, $C(CH_3)_2$], 5.66 (1H, d, $^3J_{CH=CH}$ = 10.5 Hz, 3'-H), 6.65 (1H, d, $^3J_{CH=CH}$ =10.5 Hz, 4'-H), 6.94-8.20 ppm (8H, m, aromatic).

UV spectrum (in 2-propanol), λ_{max}, nm (log ε): IIIa 297 sh (3.29), 304 (3.52), 313 (3.24); IIIb 301 (3.73), 315 (3.87), 341 (3.60), 355 (3.63); IIIc 305 (3.84).

In the case of irradiation with UV light (with a DRSh-250 Hg lamp with λ_{max} 313 nm under steady-state conditions for 10-30 sec) solutions of spiropyrans III in 2-propanol (c ~ $5 \cdot 10^{-5}$ mole/liter) at –50°C display reversible photochromic transformations associated with electrocyclic opening of the pyran fragment in the electronically excited state, which leads to isomerization of spirocyclic form III to valence-tautomeric o-quinoneallide form III*. The maxima of the long-wave absorption bands of the photoinduced III* forms were recorded in the electronic spectra: III* a 376 sh, 397, 511 sh; III*b 448; III*c 533 sh, 571 nm. Colored photoinduced form III*b is stable at room temperature ($\tau/2$ ~ 250 sec at 20°C).

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