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REACTION OF 6-CYANO-1,2,4-TRIAZOLO[1,5-*a*]PYRIMIDIN-5(8H)-ONE WITH PHOSPHORUS OXYCHLORIDE IN THE PRESENCE OF N,N-DIETHYLANILINE

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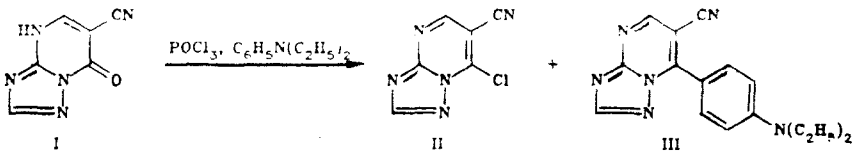
The interaction of the oxy derivatives of nitrogen heterocycles with $POCl_3$ in the presence of N,N-dialkylanilines is often utilized for the conversion of these compounds to the corresponding chlorine derivatives. However, this reaction sometimes proceeds ambiguously. For example, the reaction of some pyrimidinediones with $POCl_3$ in the presence of N,N-dimethylaniline leads to the formation of the mixture of dichloropyrimidines and N-methylanilinopyrimidines [1].

We found that the boiling of 6-cyano-1,2,4-triazolo[1,5-*a*]pyrimidin-5(8H)-one (I) in the mixture of $POCl_3$ and N,N-diethylaniline leads to the unexpected formation of 5-(p-diethylamino)phenyl-6-cyano-1,2,4-triazolo[1,5-*a*]pyrimidine (III) together with the 5-chloro derivative (II):



I II III

The mixture of 1.2 g (7.5 mmoles) of compound (I) [2], 26 ml of $POCl_3$, and 2.4 ml (2.25 g; 15 mmoles) of N,N-diethylaniline is boiled for 2 h. The excess of the $POCl_3$ is distilled off in vacuo, and the residue is poured onto ice. The residue is filtered off and recrystallized prior to the isolation of 0.24 g (18%) of compound (II), which has mp 170°C (from ethyl acetate). PMR spectrum (in CF_3COOD): 8.8 (1H, s, 7-H) and 8.93 ppm (1H, s, 2-H). The aqueous filtrate is extracted with chloroform. The extract is washed with water, dried with $MgSO_4$, and filtered. The chloroform is distilled off, and the residue is chromatographed on a column with silica gel; the fraction with the R_f 0.2 (chloroform) is collected. The yield of compound (III) is 0.44 g (20%); mp 174.5-177°C from ethyl acetate. IR spectrum (KBr): 2222 cm^{-1} (CN). PMR spectrum (CF_3COOD): 0.91 (6H, t, CH_3), 3.54 (4H, q, CH_2), 7.55 (2H, d, arom. prot.), 7.93 (2H, d, arom. prot.), 8.8 (1H, s, 7-H), and 9.1 ppm (1H, s, 2-H). Mass spectrum (m/z) (I_{rel}, %): M^+ 292 (28), $[M - CH_3]^+$ 277 (100), and $[M - CH_3{-}C_2H_4]^+$ 249 (47).

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