LETTERS TO THE EDITOR

INTRAMOLECULAR CYCLIZATION OF 1-CHLORO-3-METHYL-11-HYDROXY-ANTHRAPYRIDONE

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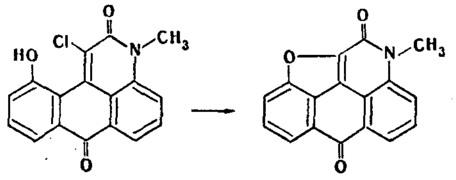
UDC 547.838'722

When 0.005 mole of 1-chloro-3-methyl-11-hydroxyanthrapyridone (I) (1-chloro-3-methyl-11-hydroxydibenzo[f,i,j]isoquinoline-2,7-dione) is heated in 30 ml of dimethylformamide or dimethylsulfoxide at 145°C for 20 min, it undergoes intramolecular cyclization to give 1 g (82%) of yellow crystals of 2-methyl-6H-furo[3,4,5-d,e]dibenzo[f,i,j]isoquinoline-1,6-dione (II) with mp 331-332°C (from nitrobenzene).



I II

Starting I was obtained in 94% yield by treatment of 1.62 g of 1-chloroacetylmethylamino-8-hydroxyanthraquinone with 5 ml of 20% NaOH; after 20 min, the reaction mixture was poured into water, the aqueous mixture was neutralized with acetic acid, and the precipitate was removed by filtration and washed with water to give 1.44 g of yellow crystals of I with mp 179-182°C (from toluene). The results of elementary and mass-spectral analysis were in agreement with the calculated values.

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6,7-CYCLOPENTANO-1,4-PERHYDROOXAZEPINE DERIVATIVES

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We have established that 2-methylcyclopentane-1,3-dione (Ia) undergoes amidoalkylation with $BzNHCH_2OH$ and BF_3•etherate to give a 2-benzamidomethylene derivative (Ib) (60%) with mp 223-225°C (from methanol), which undergoes ketalization to give an ethyleneketal (Ic) (90%) with mp 119-121°C (from methanol). The reduction of Ic with sodium borohydride in ethanol at −78°C proceeds stereospecifically to give a hydroxy amide (IIa) (88%) with mp 63-65°C (from petroleum ether). A distinct absorption band at 3473 cm^{-1}, which is characteristic for the presence in IIa of a strong intramolecular hydrogen bond, appears in the IR spectrum of IIa in $CHCl_3$ ($7.7 \cdot 10^{-4}$ mole/liter); this indicates a cis orientation of the OH and CH_2NHBz groups. Treatment of hydroxy amide IIa with NaH and $ClCH_2COOCH_3$ in tetrahydrofuran converted it to an amido ester (IIb) (90%) in the form of an oil, which was readily cyclized to give a lactam (IIIa) (95%), with mp 88-90°C (from methanol), when it was refluxed with $KHSO_4$ in xylene. Reduction of lactam IIIa with lithium aluminum hydride in refluxing dioxane and subsequent hydrolysis with aqueous hydrochloric acid in dioxane gave the N-benzoyloxazepine derivative (IIIb) in 55% yield as an oil, which was converted to the corresponding hydroxy

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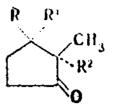
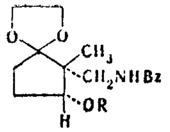
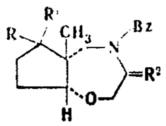
compound (IIIc) (80%), with mp 38-40°C (from methanol), by reduction with lithium aluminum hydride. Compound IIIc was characterized in the form of the 3,5-dinitrobenzoate with mp 120-121°C (from ether).



I a-c



II a,b



III a-c

Ia R+R¹=O, R²=H; b R+R¹=O R²=CH₂NHBz; c R+R¹=OCH₂CH₂O, R²=CH₂NHBz; IIa R=H; b R=CH₂COOCH₃; IIIa R+R¹=OCH₂CH₂O, R²=O; b R+R¹=O, R²=H₂; c R=OH, R¹=H, R²=H₂

The structures of the compounds obtained were confirmed by the IR, PMR, and mass-spectral data. The results of elementary analysis of Ib, c, IIa-c, and IIIa-c were in agreement with the calculated values.

SYNTHESIS OF IMIDAZO[1,2-b]ACENAPHTHENO[1,2-e]-1,2,4-TRIAZINES

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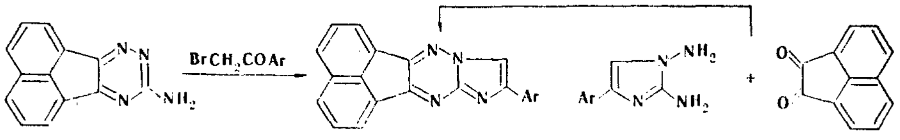
UDC 547.785.5'873.07

We have established that 2-aryl-substituted imidazo[1,2-b]acenaphtheno[1,2-e]-1,2,4-triazines (II) are formed when 3-aminoacenaphtheno[1,2-e]-1,2,4-triazine (I) is heated with α-halo ketones in refluxing organic solvents (CH_3COOH, butanol). This polyheterocyclic system was previously unknown. The following data are given for the synthesized compounds [melting points, R_f values in a methanol–butanol–water system (25:2:30), UV spectra (dimethylformamide), λ_{max} (log ε), and yields in percent]: IIa, 283-284, 0.85, 320.5 (4.53), 324 (4.49), 432 (4.33), 60; IIb, 366-367, 0.66, 320 (4.28), 340 (4.24), 430 (4.07), 69; IIc, 353-355, 0.60, 320.5 (4.48), 342 (4.45), 431 (4.26), 71.

The structure of II was confirmed by synthesis from substituted 1,2-diaminoimidazole (III) and acenaphthenequinone (IV) by refluxing in butanol for 1.5-2 h. The yields of IIa, b, c were 50, 78, and 87%, respectively.



I II a-c III IV

IIa Ar=C_6H_5; b Ar=p-BrC_6H_4; c Ar=p-ClC_6H_4

The results of complete elementary analysis of II were in good agreement with the calculated values.

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