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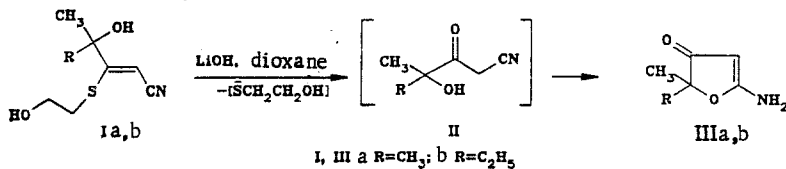
CYCLIZATION OF 3-HYDROXY-3-ALKYL-2-(2-HYDROXYETHYLTHIO)-1-BUTENECARBONITRILES TO 5-AMINOFURAN-3-ONES

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3-Hydroxy-3-methyl-2-propylthio-1-butenecarbonitrile, obtained by the reaction between tertiary cyanoacetylenic alcohols and propanethiol, is highly stable, and does not undergo changes (isomerization or cyclization) either on keeping or on heating [1].

We have found that the sulfides (I), obtained from the same cyanoacetylenic alcohols and 2-mercaptoethanol, are very different in their chemical properties and undergo intramolecular cyclization to give, instead of the expected 1,3-oxathiolanes or iminodihydrofurans [2, 3], the 5-amino-2,2-dialkyl-2,3-dihydrofuran-3-ones (III).



An intermediate in the conversion of (I) into (III) appears to be the hydroxyketonitrile (II), formed by hydrolytic cleavage of the sulfide moiety. A similar cyclization has been observed when (I) is kept at room temperature (50 days).

5-Amino-2,3-dihydro-2,2-dimethylfuran-3-one (IIIa). A mixture of 0.3 g (1.6 mmole) of the sulfide (Ia), 0.03 g of LiOH, and 6 ml of dioxane was stirred for 3 h at 50°C. The dioxane was then removed under reduced pressure, and the residue dissolved in acetone and the solution filtered. Removal of the acetone gave 0.1 g (50%) of the aminofuranone (IIIa), mp 228-230°C (sublimes). ¹H NMR spectrum (DMSO-D₆), δ: 7.76 (2H, s), 4.28 (1H, s), 1.29 ppm (6H, s). ¹³C NMR spectrum (DMSO-D₆): 178.29 [C₍₅₎], 87.87 [C₍₄₎], 198.17 [C₍₃₎], 75.79 [C₍₁₎], 23.51 ppm (CH₃). IR spectrum (chloroform): 980...1050 (C-O-C), 1600 (C=CH), 1680 (C=O), 3420, 3450 cm⁻¹ (NH₂).

5-Amino-2,3-dihydro-2-methyl-2-ethylfuran-3-one (IIIb) was obtained similarly in 68% yield, mp 205-206°C (sublimes). PMR spectrum (DMSO-D₆), δ: 7.80 (2H, s), 4.30 (1H, s), 1.50 (2H, q), 1.30 (1H, s), 0.99 ppm (3H, t). IR spectrum (chloroform): 980...1070 (C-O-C), 1600 (C=CH), 1680 (C=O), 3420, 3450 cm⁻¹ (NH₂).

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