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SHORT COMMUNICATIONS

Cyclization of 3-Alkylamino-4-hydroxybutanamides in Alkaline Medium

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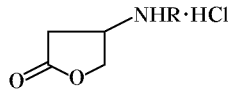
We previously developed a procedure for preparation of polyfunctional 3-alkylamino-4-hydroxybutanamides **I** [1]. Their structure implies the possibility for intramolecular cyclization which can involve either deamination with formation of substituted lactone **II** or dehydration to give 2-pyrrolidinone **III** (Scheme 1).

The transformation **I** → **II** can be effected by hydrolysis of the amido group and subsequent intramolecular cyclization of 4-hydroxybutanoic acid which should readily lose water to give the corresponding γ-lactone; this reaction is typical of such acids [2]. The transformation of **I** into pyrrolidinone **III** should involve dehydration via binding of water by chemical means or thermolysis.

We performed alkaline hydrolysis of compounds **I** (R = $CH_2C_6H_5$, C_4H_9) in alcoholic medium and obtained crystalline products which were identified as 4-benzylamino- and 4-butylaminofuran-2-one hydrochlorides **IVa** and **IVb**.



IVa, IVb

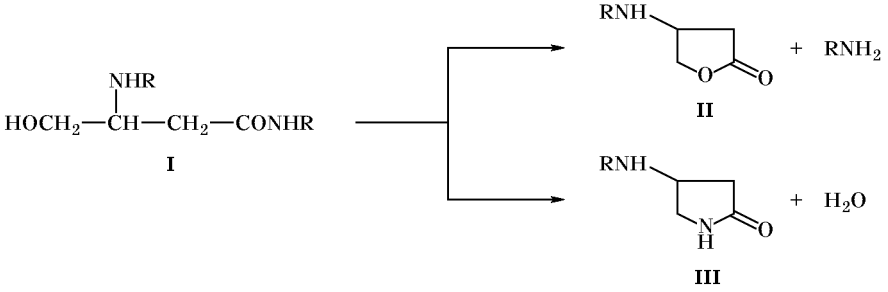
R = $C_6H_5CH_2$ (**a**), C_4H_9 (**b**).

Amides are known to undergo hydrolysis in the presence of both acid and base catalysts; as a rule, the hydrolysis is irreversible [3]. Our attempts to isolate substituted pyrrolidinone were unsuccessful. Presumably, it is not formed under the given conditions due to the presence of excess alkali.

The IR spectrum of hydrochloride **IVa** contained a band at 1775 cm^{-1} which is typical of carbonyl group in saturated lactones. The corresponding band in the IR spectrum of **I** was observed at 1640 cm^{-1}.

4-Benzylaminofuran-2-one hydrochloride (IVa). A solution of 2.5 mmol of potassium hydroxide in ethanol was added with stirring to a heated solution of 1 mmol of *N*-benzyl-3-benzylamino-4-hydroxybutanamide in ethanol. The substrate consumption was monitored by TLC. After cooling, the mixture was acidified with hydrochloric acid to pH 3–5. The organic phase was diluted with ethyl acetate–diethyl ether (1:1), the precipitate was filtered off, and the filtrate was dried and evaporated under reduced pressure. The oily residue crystallized on addition of ethyl acetate. The precipitate was filtered off. Yield 73%. Colorless crystals, mp 180–181°C. IR spectrum, ν, cm^{-1}: 1140, 1355, 1430 (C–O–C), 1575 (δNH_2^+), 1775 (C=O), 2405 (NH_2^+). Found, %: C 58.06; H 6.24; Cl 15.71; N 6.10. $C_{11}H_{14}ClNO_2$. Calculated, %: C 58.02; H 6.15; Cl 15.60; N 6.15.

Scheme 1.



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4-Butylaminofuran-2-one hydrochloride (IVb) was synthesized in a similar way. Yield 64%. mp 168–170°C. IR spectrum, ν, cm^{-1}: 1145, 1365, 1440 (C–O–C), 1565 (NH_2^+), 1785 (C=O), 2410, 2440 (δNH_2^+). Found, %: C 49.58; H 8.19; Cl 18.33; N 7.29. $C_8H_{16}ClNO_2$. Calculated, %: C 49.61; H 8.27; Cl 18.35; N 7.24.

The IR spectra of compounds **IVa** and **IVb** dispersed in mineral oil were recorded on a UR-20 spectrometer.

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