

LETTERS TO THE EDITOR

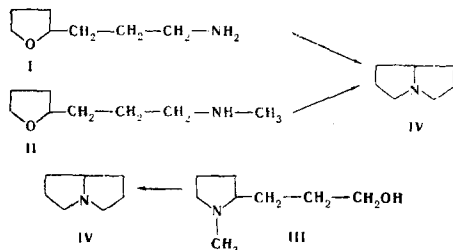
A STUDY OF THE REACTION MECHANISM OF THE CATALYTIC INTRAMOLECULAR CONVERSION OF HETEROCYCLES

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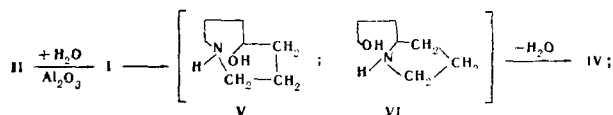
Kimiya Geterotsiklicheskikh Soedinenii, Vol. 3, No. 6, p. 1127, 1967

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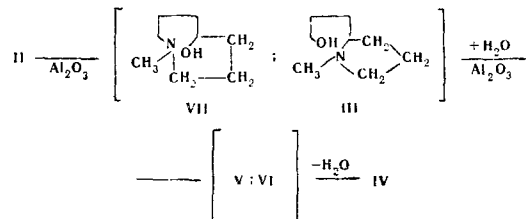
In order to study the reaction mechanism of the catalytic intramolecular conversion of heterocycles [1-3] and to broaden the field of their application, we have studied the dehydration over Al_2O_3 of 1-amino-3-(α -tetrahydrofuryl) propane (I), 1-methylamino-1-(α -tetrahydrofuryl) propane (II), and 2-(3-hydroxy-1-propyl)-1-methylpyrrolidine (III), leading in all three cases to pyrrolizidine (IV).



Bearing in mind the general laws given by Yu. K. Yur'ev [4], it is possible to envisage the formation of IV from II by two possible routes which do not exclude one another: 1) the conversion of II into I by catalytic demethylation of the secondary amino group with subsequent dehydration to IV through either or both of V and VI.



2) the catalytic isomerization of II into VII and III and their demethylation to V and VI with subsequent dehydration to IV.



To confirm the possibility of the second route via III, we have subjected the latter to dehydration. Of course, the successful dehydration of III does not exclude the variant $II \rightarrow [VII] \rightarrow [V] \rightarrow IV$. The choice between the first and second routes requires further study.

IV (from I). Yield 65.9%. Bp 70.5-71.5° C (60 mm), 97-99° C (161 mm), 145-146.5° C (765 mm), d_4^{20} 0.9149; n_D^{20} 1.4700^{5,6}. Found, %: C 75.41; 5.64; H 11.86; 12.09%; MR_D 33.90. Calculated for $C_7H_{13}N$, %: C 75.61; H 11.79; MR_D 34.07. Picrate: mp 257.5-259° C (decomp, from ethanol) [5, 6], Found, %: N 16.00; 16.03. Calculated for $C_7H_{13}N \cdot C_6H_2(NO_2)_3OH$, %: N 16.47. Compound IV was obtained from II with a yield of 58.6% and from III with a yield of 51.6%.

The IR spectrum of the samples of IV obtained from I, II, and III were identical and the picrates were identical (absence of a depression of the melting point in mixed samples).

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THE PREPARATION OF 1,3,4-OXADIAZOLES

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We have found that 2,5-dialkyl-1,3,4-oxadiazoles, which are difficult of access and have scarcely been studied, can be obtained in a single stage from carboxylic acids and hydrazine hydrochloride by heating them in boiling phosphorus oxychloride with subsequent

fractional distillation of the reaction mixture.

