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# REACTION OF 1-METHYLTHIO-3,3-DIMETHYL-3,4-DIHYDROISOQUINOLINE WITH GLYCINE

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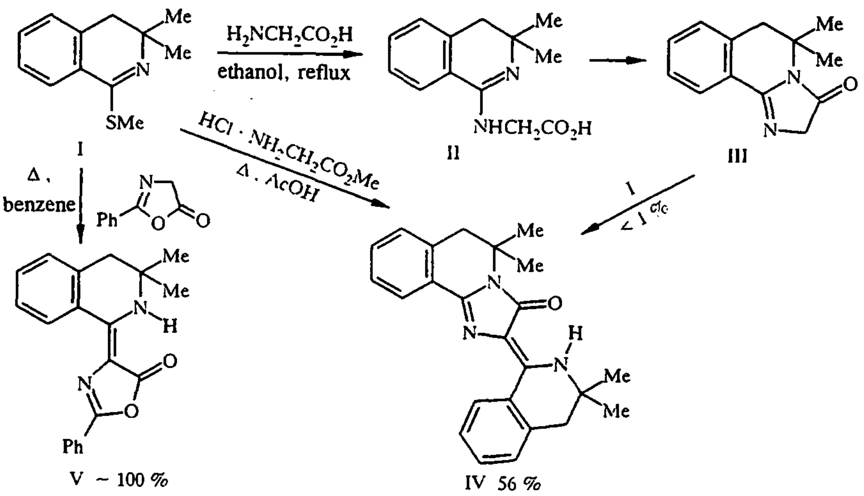
We have previously reported the synthesis of amidinoacids such as II obtained as the single products of the reaction of thiolactimic ether I with amino acids [1]. A more detailed study of the reaction of thioether I with glycine revealed the formation of 2-(3,3-dimethyl-1,2,3,4-tetrahydro-1-isoquinolylidene)-5,5-dimethyl-2,3,5,6-tetrahydroimidazo[2,1-*a*] isoquinolin-3-one (IV) in a yield of less than 1%. Product IV is probably the product of a secondary reaction of thioether I and intermediate imidazoisoquinoline III. Indeed, the reaction of thioether I with a model compound, namely, 2-phenyl-5-oxazolone in benzene at reflux gave V in quantitative yield. Product IV was obtained in satisfactory yield upon the reaction of two equivalents of thioether I with the hydrochloride salt of the methyl ester of glycine in acetic acid at reflux.



The PMR signals for the 8′-H proton in IV and 8-H proton in V are shifted downfield by more than 1 ppm relative to their position in previously studied isoquinolines with an exocyclic C=C bond or carbon–heteroatom double bond [2]. The greater deshielding of these protons may be the result of the spatial approximation of the carbonyl group in the case of *E* configuration or of the azomethine group in the case of *Z* configuration. The reduction in the frequency of the IR stretching band of the lactam and lactone carbonyl groups as well as finding the signals for the NH protons at 9-10 ppm are evidence for the *Z* configuration with intramolecular hydrogen bonding between the N–H and C=O groups.

**2-(3,3-Dimethyl-1,2,3,4-tetrahydro-1-isoquinolylidene)-5,5-dimethyl-2,3,5,6-tetrahydroimidazo[2,1-*a*]-isoquinolin-3-one (IV)**, mp 206-208°C (from methanol). Mass spectrum, *m/z* (relative intensity, %): $[M + 1]^+$ 372 (82), $[M - CH_3]^+$ 357 (77), $[M - 2CH_3]^+$ 341 (12). PMR spectrum in $CDCl_3$: 1.37 (6H, s, 3-$CH_3$), 1.69 (6H, s, 4-$CH_3$), 2.89 (2H, s, 4-H), 2.96 (2H, s, 5-H), 7.1-7.6 (6H, m, $H_{arom}$), 8.15 (1H, m, 10-H), 9.38 (1H, m, 8′-H), 10.42 ppm (1H, br.s, NH). IR spectrum in chloroform: 3280 (N–H), 1626 (C=O), 1603 $cm^{-1}$. UV spectrum in ethanol, $\lambda_{max}$ (log $\varepsilon$): 204 (4.46), 273 (4.00), 314 (3.87), 425 nm (4.37).

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**1-(5-Oxo-2-phenyl-4-oxazolylidene)-3,3-dimethyl-1,2,3,4-tetrahydroisoquinoline (V)**, mp 161-164°C (from 2-propanol). PMR spectrum in $CDCl_3$: 1.36 (6H, s, $3\text{-}CH_3$), 2.93 (2H, s, 4-H), 7.43 (6H, m, $H_{arom}$), 8.03 (2H, m, 2″- and 6″-H), 9.19 ppm (2H, m, NH and 8-H). IR spectrum in chloroform: 3410 (N–H), 1700 (C=O), 1615 $cm^{-1}$. UV spectrum in ethanol, $\lambda_{max}$ (log $\varepsilon$): 406 nm (4.35).

The elemental analysis data for C, H, and N were in accord with the calculated values.

## REFERENCES

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