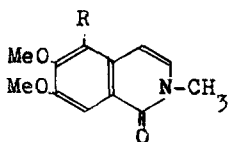


THALACTAMINE, 1-OXO-2-METHYL-5,6,7-TRIMETHOXY-
I,2-DIHYDROISOQUINOLINE IN A THALICTRUM MINUS
VARIETY

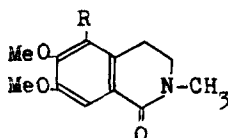
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From the above-ground parts of a *Thalictrum minus* variety spread near the Black sea coast of Bulgaria a new weakly basic alkaloid, thalactamine with molecular formula $C_{13}H_{15}NO_4$ and m.p. 112-114°C was isolated. Our attempts to elucidate the structure of this compound lead to 1-oxo-2-methyl-5,6,7-trimethoxy-1,2-dihydroisoquinoline, the first example of a new type of simple isoquinoline alkaloids.



I, R=OMe
III, R=H



II, R=H
IV, R=OMe

The mass spectrum of I shows a molecular peak at 249 m/e. The bands at 1660 cm^{-1} and 1620 cm^{-1} in the IR-spectrum are ascribed to a lactam carbonyl group and a conjugated double bond respectively (I). In good agreement with structure I is the NMR spectrum of the compound (2). The absorption of the N-methyl group at $6,46\tau$ is shifted downfield by the neighbouring carbonyl group and the double bond. There are peaks for three methoxyl groups at $6,07\tau$ from 6 protons and at $6,10\tau$ from three protons. The AB type quartet at $3,09\tau$ and $3,42\tau$ ($J=12$ cps) indicates the presence of the protons at C_3-C_4 double bond. The C_8 aromatic proton at $2,44\tau$ is shifted downfield because of the deshielding effect of the peri carbonyl group.

The close similarity between the UV, IR and NMR spectral data of I and III is also in good agreement with structure I. Compound III was obtained by Pd/C dehydrogenation of II, which was earlier prepared by $KMnO_4$ in acetone oxidation of the alkaloids

thalicarpine and thalmetatine (3). The molecular weight of III determined by MS is 219. There is absorption at 1650 cm^{-1} and 1630 cm^{-1} for the carbonyl group and the conjugated double bond in the IR-spectrum of III. The NMR spectrum of III shows the N-methyl group protons at $6,46\tau$ and C_8 aromatic proton at $2,25\tau$, shifted downfield as in I. The AB type quartet for the C_3-C_4 double bond protons appears at $3,12$ and $3,72\tau$ ($J=12\text{ cps}$). The difference between I and III is as it should be expected the absorption of two methoxyl groups at $6,07$ and $6,09\tau$ and the appearance of the C_5 aromatic proton at $3,24\tau$. The maxima in the UV spectra of I and III are at $247\text{ m}\mu$ ($\epsilon_I=42\ 000$, $\epsilon_{III}=36\ 200$); $270\text{ m}\mu$ ($\epsilon_I=4\ 000$, $\epsilon_{III}=5\ 600$); $281\text{ m}\mu$ ($\epsilon_I=4\ 800$, $\epsilon_{III}=6\ 000$); $293\text{ m}\mu$ ($\epsilon_I=5\ 500$, $\epsilon_{III}=6\ 400$) for both compounds. Differences are only in the maxima at $332\text{ m}\mu$ ($\epsilon=3\ 000$) for I and $322\text{ m}\mu$ ($\epsilon=3\ 200$) for III.

Compound I is rather stable. Acid and alkaline hydrolysis failed. The attempts to hydrogenate I with LiAlH_4 , NaBH_4 and with Adam's catalyst up to 100°C were unsuccessful. The C_3-C_4 double bond can be hydrogenated under high temperature and high pressure (80 atm. and 80°C). The resulting compound IV with m.p. $104-106^\circ\text{C}$ is completely identical according to IR and NMR spectra (TLC did not separate I and IV) with the same compound synthesised by us. The synthesis of IV was realised through 2,3,4-trimethoxyphenethylamine (4) following by formaldehyde/formic acid cyclisation to 2-methyl-5,6,7-trimethoxy-1,2,3,4-tetrahydroisoquinoline and KMnO_4 in acetone oxidation to IV. Zinc dust distillation of I indicated by means of TLC the presence of isoquinoline.

REFERENCES

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