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SHORT COMMUNICATION

The Isolation of Methylcorypalline from Embryo Loti

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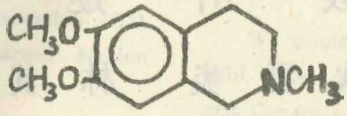
Previously, two alkaloids isoliensinine[1] and lotusine[2] had been isolated from Formosan "Lien Tze Hsin", embryo loti (embryo of *Nelumbo nucifera Gaertn.*, Fam. Nymphaeaceae). The present communication deals with the further study on the isolation and characterization of a new minor Base-A which was clarified as methylcorypalline together with neferine[3]. The concentrated ethanol extract of loti embryo was separated into alkaline soluble phenolic and insoluble non-phenolic fraction. The latter non-phenolic portion was chromatographed on alumina (Wako). Elution with benzene yielded Base-A; subsequent elution with a mixture of benzene and chloroform (1 : 1) gave neferine.



(I)

Base-A was crystallized as white feathery crystals form n-hexane, $C_{12}H_{17}O_2N$ $\frac{1}{2}H_2O$, mp. 58-59°, $[\alpha]_D^{20}$ ±0° ($CHCl_3$), negative to $FeCl_3$ and Labat test. Several crystalline derivatives had been prepared as the hydrochloride, mp. 216-217°, picrate, mp. 152-153°, and methiodide, $C_{12}H_{17}O_2N$ CH_3I 1/3 CH_3COCH_3, mp. 242-243°.

The UV spectrum of Base-A showed a characteristic maximum absorption at 290$m\mu$ (sh.) (log. ε, 3.39), 295 (3.47) indicating more than just simple benzenoid. The IR broad band at 3250cm^{-1} indicated

the presence of hydroxyl group of semihydrate. The NMR spectrum exhibited three protons singlet of one *N*-methyl peak at 7.85τ, six protons singlet of two O-methyl peak at 6.18τ, and two one proton singlets of aromatic protons at 3.40 and 3.48τ. The mass spectrum gave a molecular ion peak at *m/e* 207 ($C_{12}H_{17}O_2N$). The base peak at *m/e* 164 (207—C_2H_5N) and other intense peaks were found at *m/e* 206, 121, 91, and 76. These spectroscopic and analytical data suggest that this base is a dimenthoxy-*N*-methyl-tetrahydroisoquinoline derivatives, most likely comprising two methoxy group at C_6 and C_7.

In order to confirm this assumption, a six step synthesis of 6, 7-dimethoxy-2-methyl-1, 2, 3, 4-tetrahydroisoquinoline (I) was developed begining with vanillin which was converted to β-(3, 4-dimethoxyphenyl) ethylamine via veratraldehyde and 3, 4-dimethoxy-ω-nitrostyrene by known methods. According to the method described by J. S. Buck[4], the 6, 7-dimethoxy-1, 2, 3, 4-tetrahydroisoquinoline was prepared by formylation of this β-(3,4-dimethoxyphenyl) ethylamine with formalin and cyclization with c-HCl. *N*-methylation of 6, 7-dimethoxy-1, 2, 3, 4-tetrahydroisoquinoline, by treating with formic acid and formalin yielded compound (I). The resulting product (I), mp. 58-59°, was completely identical to IR (nujol) spectrum and tlc with Base-A. In addition, there was no depression of the mp. when the two were admixed, confirming the identity of the synthesized compound (I) and Base-A. On the basis of these experimental evidences, the structure of Base-A is proved to be 6, 7-dimethoxy-2-methyl-1, 2, 3, 4-tetrahydroisoqinoline[5] (I). This is the first isolation of methylcorypalline (I) from natural sources.

Acknowledgement

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