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TOTAL SYNTHESIS OF (±) - DECALINE - AN ALKALOID OF LYTHRACEAE SPECIES

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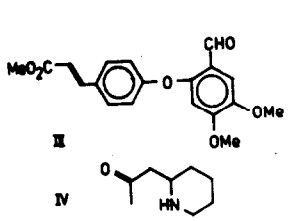
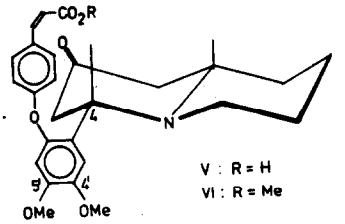
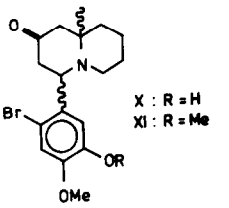
Decaline is a Lythraceae alkaloid of biphenyl ether structure[1]. The Ullmann reaction of 6-bromoveratral (I) with the methyl p-hydroxycinnamate (II) leads to biphenyl ether derivative (III). Compound (III) was isolated by chromatography on silica gel as crystalline solid of m.p. 130-131°C from benzene; the yield is 55%. (III) undergoes an alkaline condensation with isopelletierine (IV) resulting in a mixture (50% yield) of stereoisomeric aminoacids (V).(V) was an amorphous solid. Esterification of (V) with $(CH_3O)_2SO_2$ and chromatography on silica gel resulted in isolation of quinolizidine derivative with trans ring fusion and with the equatorial substituent at C-4 (VI, m.p. 112-115°C). The yield of (VI) was 70%. (VI) was synthesized in another way; the reaction of 6-bromoisovanilline (IX) with isopelletierine (IV) resulted in (X). (X) was methylated with $(CH_3O)_2SO_2$ and the quinolizidine derivative (XI^a, m.p. 138,5-140,5°C) with trans ring fusion and with the equatorial phenyl group at C-4 was isolated by chromatography on silica gel (benzene: $MeCO_2Et$, 4:1 and 3:1).



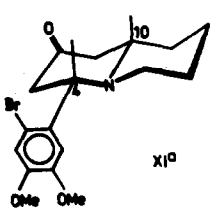




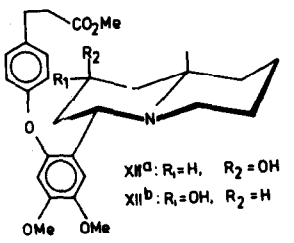
The Ullmann reaction of compound (XI^a) with (II) in pyridine resulted in the compound (VI). The catalytic reduction of (VI) on PtO_2 results in a mixture of axial (XII^a) and equatorial (XII^b) hydroxy esters in 4:1 ratio.

(XII^a) and (XII^b) were isolated by chromatography on silica gel (ethyl acetate: ethanol, 9:1) or on alumina act.III; both compounds were amorphous.

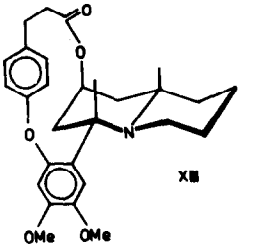
Hydrolysis of (XII^a) and cyclisation with $SOCl_2$ in chloroform solution /2/,/3/ resulted in racemic decaline (XIII), m.p. 192-194°C (methanol) identical with the original alkaloid (TLC,IR,NMR,MS).



XI^a



XII^a: R_1=H, R_2=OH
XII^b: R_1=OH, R_2=H



XIII

Reported structures and their conformations were confirmed by IR, NMR and mass spectra; satisfactory elemental analyses were obtained for all compounds. /4/

References

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