and of *N. oleander*,[3] seeds of *N. oleander*,[4] and on cyclitols of roots.[5]

Present work. Acetophenones were obtained from the ether extracts of the root-bark or heartwood on silica gel column chromatography with benzene-acetone as eluting solvent. 2,4-*Dihydroxyacetophenone:* m.p. 148–149° (0·0013% of dried bark and 0·0001% of heartwood freed from bark; m.m.p., IR, TLC). 4-*Hydroxyacetophenone:* m.p. 108–110° (0·0005% of dried bark; m.m.p., IR, TLC).

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Key Word Index—Nerium odorum; Apocynaceae; 4-hydroxy-acetophenone; 2,4-dihydroxyacetophenone.

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BUXACEAE

ALKALOIDS OF *BUXUS WALLICHIANA*

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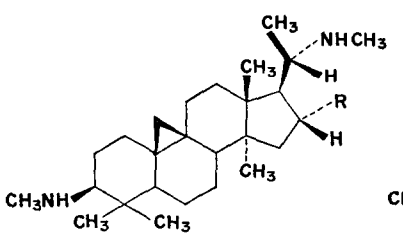
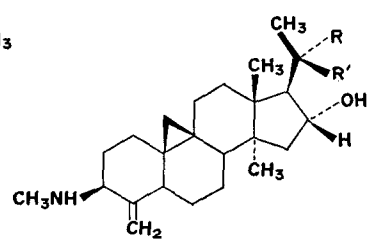
Plant. Buxus wallichiana Baill[1] from India. *Previous extraction.* None but species of *Buxus* are normally sources of steroidal alkaloids.[2]

Extraction and isolation. Ground dried leaves (4·2 kg) of *B. wallichiana* were extracted by percolation with MeOH at room temp. Evaporation left a black gum which was taken up in 2% aqu. HCl and the neutral materials (19 g) removed by continuous $CHCl_3$ extraction.



(I) R = OH
(II) R = H



(III) R = $NHCH_3$; R′ = H
(IV) R′: R = O

[1] We thank Dr. W. I. TAYLOR (International Flavors and Fragrances, N.J.) for the plant material and the Bronx Botanical Gardens, N.Y. for the identification.
[2] V. CERNY and F. SORM, *Steroid Alkaloids in the Alkaloids* (edited by R. H. F. MANSKE), Vol. IV, p. 305, Academic Press, New York (1967).

The aqueous sol. was basified (NH_4OH) and the crude base (41 g) obtained by further $CHCl_3$ extraction. Distribution of the crude base between $CHCl_3$ and aqueous acetate buffer (pH 5·6) gave three fractions: Stronger bases (advanced with acetate) 9·01 g; Intermediate bases 6·72 g; and Weaker bases (left behind in $CHCl_3$) 18·66 g. The stronger bases (8·0 g) were chromatographed over Al_2O_3 (Woelm Act. III) affording the three bases: cyclovirobuxine-D (I, 360 mg), cyclobuxine-D (II, 510 mg), and cycloprotobuxine-C (III, 104 mg). Similar treatment of the intermediate bases afforded buxtauine (IV, 456 mg) but the weaker bases gave no tractable material.

Identification. The combination of elemental analysis, interpretation of IR, NMR and MS data[3] and the preparation of suitable (previously described) derivatives was found to be effective in positively identifying the alkaloids.

Cyclovirobuxine-D (I). From acetone, m.p. 219–220° (Lit.[4] 221–224°). (Found: C, 77·6; H, 11·4; N, 7·0; O, 4·1. Calc. for $C_{26}H_{46}N_2O$: C, 77·6; H, 11·5; N, 7·0; O, 4·0%.) Eschweiler–Clarke *N*-methylation afforded cyclovirobuxine-A, m.p. 228–230°. Mixed with an authentic sample (from Dr. S. M. Kupchan) our cyclovirobuxine-D showed no depression in the m.p. TLC and IR results were identical. *Cyclobuxine-D* (II). From benzene, m.p. 239–240° (decomp. Lit.[5] 245–247°). (Found: C, 77·8; H, 10·9; N, 7·3; O, 4·0 Calc. for $C_{25}H_{42}N_2O$: C, 77·7; H, 10·9; N, 7·3; O, 4·1%.) The dimethiodide, m.p. 225–228° (lit.[6] 234° decomp.) was prepared by refluxing the base in acetone with excess CH_3I. *Cycloprotobuxine-C* (III). From acetone, m.p. 191–192° (Lit.[7] 200–202°). Eschweiler–Clarke methylation afforded cycloprotobuxine-A, m.p. 205–206° (Lit.[7] 206–207°). (Found: C, 87·0; H, 12·0; N, 6·8. Calc. for C_{28} $H_{50}N_2$: C, 87·1; H, 12·1; N, 6·8%.) *Buxtauine* (IV). From acetone m.p. 179–180° (Lit.[8] 181–183°). (Found: C, 77·6; H, 10·2; N, 3·8; O, 8·6. Calc. for $C_{24}H_{37}NO_2$: C, 77·6; H, 10·0; N, 3·8; O, 8·6%.) Acetylation afforded the O,N-diacetyl derivative, m.p. 208–210° (Lit.[8] 211–213°).

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Key Word Index—Buxus wallichiana; Buxaceae; steroidal alkaloids; cyclovirobuxine D; cyclobuxine D; cycloprotobuxine C.