connected to the outlet of the column and cooled in a mixture of dry ice-acetone. The condensed headspace vapours were then injected into the gas chromatograph.

Gas chromatography. Analytical GLC separations were carried out on a Perkin-Elmer model 900 gas chromatograph equipped with a FID and intermittently also with a capillary injector (Table 2).

MS. The MS were recorded at 70 eV on an LKB 9000 combined GC–MS apparatus equipped with a 3 m × 6 mm (o.d.) glass column filled with 7·5% SE 30 on Chromosorb W-AW-DMCS 80–100 mesh.

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ALKALOIDS OF *LYCOPODIUM THYOIDES* AND *L. CONTIGUUM*

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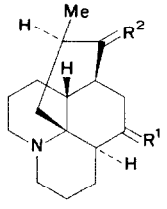
Key Word Index—*Lycopodium thyoides*; *L. contiguum*; Lycopodiaceae; lycopodine; fawcettiine; *O*-acetylfawcettiine; *O*-acetyldihydrolycopodine; clavolonine.

Plants. Lycopodium thyoides H. B. Willd. and *L. contiguum* Klotzsch. *Source.* Collected near Bogota, Columbia in June 1970 by Dr. J. H. Wilce. Voucher specimens (*L. thyoides*, No. 41359; *L. contiguum*, No. 41358) deposited in the University of Alberta Herbarium.

Present work. Ground, dried, whole plant was extracted (Soxhlet) with MeOH, concentrated, and taken up in 1% aq. HCl. The acidic soln was washed with Et_2O, basified with aq. NH_3, and extracted with $CHCl_3$. In the case of *L. thyoides* TLC (aluminum oxide G, $CHCl_3$–MeOH, 49:1, Dragendorffs reagent) indicated at least five components. These were separated by chromatography over alumina (eluent $CHCl_3$–MeOH, 49:1) and identified, in order of elution, as follows: (a) lycopodine (**1**),[1] identified as the hydrochloride, m.p. > 300°, by comparison (IR) with an authentic sample, and as the free base (IR, MS, TLC identical with an authentic sample); (b) *O*-acetylfawcettiine (**2**),[2] IR identical with that of an authentic specimen, further characterized as the methiodide, m.p. 271–272° (from MeOH), ν_{max} 1738 cm^{-1}; (c) *O*-acetyldihydrolycopodine (**3**),[3] m.p. 94–96° (lit.[4] 95–96°), hydroperchlorate, m.p. 246–247° (lit.[4] 246–247°), identified by comparison (IR, and in the case of the free base, TLC and MS) with authentic samples; (d) fawcettiine (**4**),[5] identified by comparison (IR) of its methiodide, m.p. 293–294° (from CH_2Cl_2–MeOH) (lit.[6] 296–297°) with an authentic sample. The free base was not obtained crystalline, but it showed TLC behavior identical with authentic fawcettiine; (e) the final component eluted formed

[1] MACLEAN, D. B. (1968) *The Alkaloids* (MANSKE, R. H., ed.), Vol. 10, pp. 328–334, Academic Press, New York.
[2] BURNELL, R. H. and TAYLOR, D. R. (1960) *Chem. Ind.* 1239.
[3] Ref. 1, p. 334.
[4] Ref. 1, p. 311.
[5] ANET, F. A. L. (1960) *Tetrahedron Letters* (20) 13.

a crystalline hydroperchlorate, m.p. 210–212° (from acetone), ν_{max} 3350, 1728, 1230 cm^{-1}. The MS of the non-crystalline free base showed an apparent molecular ion at *m/e* 305 ($C_{18}H_{27}O_3N$) and significant peaks at *m/e* 246, 234, 174 (base), and 146. This compound has not been identified.



(**1**) R^1 = O; R^2 = H, H

(**2**) R^1 = OAc, H; R^2 = H, OAc

(**3**) R^1 = OAc, H; R^2 = H, H

(**4**) R^1 = OAc, H; R^2 = H, OH

(**5**) R^1 = O; R^2 = H, OH

In the case of *L. contiguum*, TLC again indicated five components. These were separable by chromatography over basic alumina and identified, in order of elution, as follows: (a) lycopodine (**1**),[1] identified by comparison (TLC, IR, m.p.) with an authentic sample; (b) *O*-acetylfawcettiine (**2**),[2] identified by TLC and by comparison of the methiodide with that reported above; (c) clavolonine (**5**),[7] m.p. 237–238°, identical (IR, MS, TLC) with an authentic sample; (d) fawcettiine (**4**),[5] obtained after sublimation as a white powder, identical (IR, TLC) with authentic material. The IR spectrum of the methiodide, m.p. 293–294° (lit.[6] 296–297°), was superimposable on that of an authentic sample; (e) the most polar component showed TLC behavior and IR spectrum identical with that of the unidentified alkaloid $C_{18}H_{27}O_3N$ isolated from *L. thyoides*.

[6] ANET, F. A. L. and KHAN, N. H. (1959) *Can. J. Chem.* **37**, 1589.
[7] BURNELL, R. H. and TAYLOR, D. R. (1961) *Tetrahedron* **15**, 173.