3,4-DIMETHOXY-ω-(2'-PIPERIDYL)ACETOPHENONE, A NEW ALKALOID FROM *BOEHMERIA PLATYPHYLLA* DON. (FAMILY URTICACEAE)*

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There has been little chemical investigation of the alkaloids of plants of the family Urticaceae, and the only known alkaloids of this family appear to be 5-hydroxytryptamine from the stinging nettle, Urtica dioica L.,¹ a liquid alkaloid thought to be coniine (2-n-propylpiperidine) from Parietaria officinalis L.,² and incompletely characterized alkaloids from Trema micrantha and Trymatococcus amazonicus.³ Examination of the eastern Australian

species Boehmeria platyphylla Don. has now led to the isolation of a new alkaloid, $C_{15}H_{21}NO_3$, which has been identified as 3,4-dimethoxy- ω -(2'-piperidyl)aceto-phenone (I).

The alkaloid (I) is closely related to the alkaloid pleurospermine $(II)^4$ previously isolated from the botanically unrelated species, *Cryptocarya pleurosperma* White & Francis, belonging to the family Lauraceae, and chemical proof of the structure of (I) was obtained from its synthesis by *O*-methylation of pleurospermine. Like pleurospermine, the alkaloid (I) is optically inactive and this observation is in accord with the examples, previously discussed⁴ in relation to pleurospermine, which illustrates the ease of racemization of asymmetric carbon atoms in environments similar to that at C2'.

Experimental

B. platyphylla was collected in the Toonumbar State Forest, New South Wales, and dried whole plants (17 kg) were milled and extracted by continuous percolation with ethanol at 40°. The concentrate remaining after evaporation of most of the ethanol under reduced pressure was acidified by addition of dilute hydrochloric acid and diluted with water. After filtration, the acidified solution was brought to pH 9.0 by addition of ammonia and repeatedly extracted

* Manuscript received January 9, 1968.

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- ¹ Collier, H. O. J., and Chesher, G. B., Br. J. Pharmac. Chemother., 1956, 11, 186.
- ² Remziye Salih Hasar, Türk Ij. tecr. Biyol. Derg., 1951, 11, 172 (Chem. Abstr., 1953, 47, 1893).
- ³ Boit, H. G., "Ergebnisse der Alkaloid-Chemie bis 1960." p. 957. (Akademie-Verlag: Berlin 1961.)

⁴ Loder, J. W., Aust. J. Chem., 1962, 15, 296.

Aust. J. Chem., 1968, 21, 1397-8

with chloroform. Evaporation of the combined chloroform extracts gave a crude alkaloid fraction, which was purified by extraction from chloroform solution into dilute (2x) sulphuric acid. The combined aqueous acid extracts were basified ($\rm NH_3$) and extraction with chloroform gave the crude alkaloids (5.7 g).

When the crude alkaloids were warmed with benzene, the greater part (4.6 g) dissolved leaving an intractable black tarry residue. The benzene solution was added to a column of neutral alumina but only small amounts of material were eluted, by benzene. A mixture of benzene-chloroform (10:1) eluted a series of crystalline fractions (2.0 g) consisting essentially of the major alkaloid (I), and when these fractions were combined and crystallized from acetone, (I) was obtained as colourless crystals, m.p. $81 \cdot 5 - 82 \cdot 5^{\circ}$, $[a]_{\rm D} \pm 0^{\circ}$ (c, 2.0 in ethanol) (Found: C, 68.7; H, 8.3; N, 5.3. Calc. for $C_{15}H_{21}NO_3$: C, 68.4; H, 8.0; N, 5.3%). Alkaloid (I) had $\lambda_{\rm max}$ 275 and 304 m μ (log $\epsilon 4.05$ and 3.95 respectively), $\lambda_{\rm min}$ 292 (log $\epsilon 3.87$) in ethanol, and $\nu_{\rm max}$ (CHCl₃) 3400 cm⁻¹ (NH), 1670 (conjugated carbonyl), and 1590 (aromatic ring). The n.m.r. spectrum showed a sharp six-proton singlet at $\delta 3.90$ (two methoxyl groups), signals from three aromatic protons at $\delta 6.90$ (H 5 doublet) and 450-463 c/s (H 2, H 6), and broad multiplet signals for the other protons. The mass spectrum showed peaks at m/e 263 (M⁺; 6% of base peak), 180 (50%), 165 (base peak), 137 (10), 122 (8), 84 (45), and 83 (36).

Methylation of pleurospermine with diazomethane in ether afforded a product, m.p. $^{\circ}$ 81-82 \cdot 5°, identical with (I). The identification was confirmed by a mixed melting point determination and the identity of the infrared spectra of the two samples.

Reduction of (I) (50 mg) with sodium borohydride (50 mg) in ethanol (5 ml) gave a dihydro derivative, which crystallized from acetone as colourless prisms, m.p. $143-5^{\circ}$, λ_{\max} 228 and 277 m μ (log ϵ 3.94 and 3.48 respectively). The i.r. spectrum (CHCl₃ solution) showed no carbonyl absorption and the molecular weight was confirmed by the mass spectrum (M⁺, 265) (Found: C, 67.8; H, 8.7; N, 5.4. Calc. for C₁₅H₂₃NO₃: C, 67.9; H, 8.7; N, 5.3%).

Acknowledgments

The authors wish to thank Dr J. W. Loder for the gift of a sample of pleurospermine, and Mr W. T. Jones for the collection and identification of *Boehmeria platyphylla* Don.