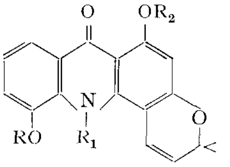
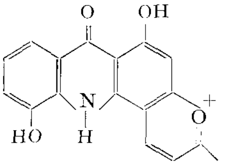
SPECIALIA

Extractives of Rutaceae: Atalaphyllidine, a New Acridone Base

Petroleum ether extract of the root bark of *Atalantia monophylla* Correâ afforded a mixture of acridone alkaloids together with a number of oxygen heterocycles[1–3]. The present communication is concerned with the elucidation of structure **1** for one of the minor bases, atalaphyllidine, $C_{18}H_{15}NO_4$ (M^+ 309), m.p. 275° (dec), λ_{max} (EtOH) 259, 270, 296, 324 and 405 nm (log ε; 4.72, 4.63, 4.43, 4.15, 3.82), absorption typical of that of 9-acridone alkaloids[4] and showed aluminium bathochromic shifts indicating the presence of hydroxy groups[5]. Its IR-spectrum showed a highly chelated CO at 1640 cm^{-1} and OH/NH at 3380 cm^{-1}. The existence of the OH function as phenolic hydroxyl was indicated by a green ferric reaction. 100 MHz NMR-spectrum of the base in C_5D_5N revealed the presence of a 2,2-dimethylchromene system by the appearance of signals at 5.55 and 6.99 δ (d, J = 10 Hz each) and 1.48 δ (6 H, s). Amongst the aromatic protons, two of them appeared as multiplets around 7.32 δ, followed by the C-8 proton as a quartet at 8.14 δ. As expected, the C-2 proton appeared upfield at 6.52 δ.



1 R = R_1 = R_2 = H
2 R = Me, R_1 = R_2 = H
3 R = R_1 = R_2 = Me



(a)

The above spectroscopic data are consistent with structure **1** for atalaphyllidine which was compatible with the presence of mass fragmentation peaks at *m/e* 309 (M^+), 294 (100%, ion a), 155, 147, 133, 77, 69 and 43.

Methylation of the alkaloid with diazomethane gave a monomethyl ether **2** which also showed positive ferric reaction, thereby confirming the presence of chelated hydroxy group. The monomethyl ether **2**, $C_{19}H_{17}NO_4$ (M^+ 323), M.p. 200° showed UV- and IR-spectra essentially similar to that of the original base. NMR-spectrum (100 MHz; $CDCl_3$: DMSO 19:1) showed a 6 proton singlet at 1.42 δ (methyl protons), 3 proton singlet at 4.04 δ (methoxyl protons), 2 doublets at 5.6 and 6.52 δ (J = 10 Hz; chromene protons). The C-2 proton appeared as a singlet at 6.12 δ and the remaining aromatic protons appeared as multiplets between 7.08–7.16 δ (2H) and 7.8 δ (1H). The NH and the hydroxy proton at C-1 appeared as singlets at 8.62 and 10.32 δ which are exchangeable with D_2O.

Prolonged treatment of atalaphyllidine **1** in dry acetone with CH_3I in presence of anhydrous K_2CO_3 furnished a trimethyl derivative **3**, m.p. 175–176° which was identical with that of dimethyl derivative of alkaloid A[5] from m.m.p and superimposable IR-spectra, thus establishing structure **1** for the new base.

Summary. The structure of acridone alkaloid atalaphyllidine, has been established as 2′,2′-dimethyl-[pyrano 5′,6′:3,4]-1,5-dihydroxy acridone, from spectroscopic data and chemical reactions.

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[1] D. Basu and S. C. Basa, J. org. Chem. *37*, 3035 (1972) and refercenes cited therein.

[2] S. C. Basa, Phytochemistry, *14*, 835 (1975).

[3] J. D. Shringarpure and B. K. Sabata, Indian J. Chem. 13, 24 (1975).

[4] J. Reisch, K. Szendrei, E. Minker and I. Novak, Pharmazie *4*, 208 (1972).

[5] A. W. Fraser and J. R. Lewis, J. chem. Soc. Perkin *1*, 1173 (1973).

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Long Alkyl Chains-3-Substituted Pyrrole-2-Aldehyde (-2-Carboxylic Acid and Methyl Ester) from the Marine Sponge *Oscarella lobularis*

In the course of our continuing search for new metabolites from marine sponges, we undertook the determination of the chemical compounds of *Oscarella lobularis*, which was found to contain a novel group of substances characterized by saturated, mono- and di-unsaturated long alkyl chains linked at position 3 of pyrrol-2-aldehyde, -2-carboxylic acid and 2-carbomethoxy moieties.

Fresh tissues of the sponge, collected in the bay of Naples, were exhaustively exctracted with acetone. Ether-water partition of the residue and chromatography on silica gel of the ether extract (2.4 g from 22 g of the dry weighted animal) gave 3 fractions, viz a) in benzene, an ester fraction (60 mg; 0.3% based on dry weight of the animal); b) in 9:1 benzene-ether, an aldehyde fraction (150 mg; 0.8%) and c) in 7:3 benzene-ether, a carboxylic acid fraction (6 mg; 0.03%).

Aldehyde fraction. Chromatography of this fraction from 12% $AgNO_3$ – impregnated silica gel in 95:5 benzene-