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# THE STRUCTURE OF OKOLASIN, A NEW ALKALOID FROM PIPER GUINEENSE

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The chemistry of the *genus* Piper has been extensively investigated[1-4]. The fruits have been shown to exhibit variation in their chemical composition dependent on geographical location[5]. Jagdev et al[6] have reported the isolation and structure elucidation of trichostachine I (R = H) from the leaves of Piper peepuloides. In the present investigation we wish to report the isolation and structure elucidation of a new alkaloid, 6-methoxy trichostachine I (R = $OCH_3$) designated as okolasin.

The petroleum ether extract of the air dried finely powdered seeds (500 g) of Piper guineense (collected in Okola-Cameroon) on chromatography over alumina yielded a fraction in benzene eluates which recrystallised from benzene/petroleum ether, m.p. 172-174° (yield : 0.05 %).

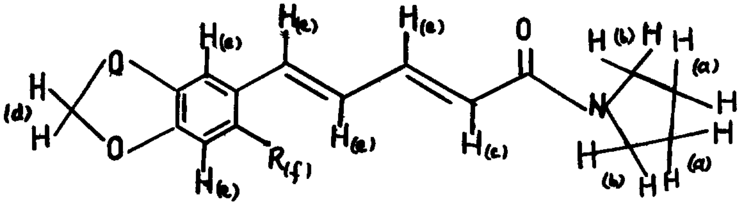
The compound analysed for $C_{17}H_{19}NO_4$, $M^+$301 ; UV, $\lambda_{max}^{EtOH}$ 203, 307, 375 $m_\mu$ exhibits a chromophoric system identical with that of piperine type amides. The IR (KBr) spectrum of the alkaloid shows principal peaks at 1592 $cm^{-1}$, 1635 $cm^{-1}$ (extended α,β -unsaturated amide), 1260 $cm^{-1}$, 1035 $cm^{-1}$ and 930 $cm^{-1}$ (methylenedioxy group), 2860 $cm^{-1}$ (methoxy group) and 1610 $cm^{-1}$ (trans configuration of olefinic double bond).

The NMR ($CDCl_3$) is consistent with structure I (R = $OCH_3$) and the position of all the protons are according to expectations.

The mass fragmentation pattern is also in agreement with the proposed structure.

Hydrolysis of the alkaloid with 20 % alcoholic KOH afforded a base and an acid which were identified as pyrrolidine and 6-methoxypiperic acid, m.p. 224°[7].

Oxidation with alkaline potassium permanganate gave 3,4-methylenedioxy-6-methoxybenzaldehyde, m.p. 110-111° (lit.[8], 111.5-112°). Further oxidation gave the corresponding acid, m.p. 146-147° (lit.[8], 148-149°) thus confirming that the methoxy group is at the 6-position.



TRICHOSTACHINE (R = H)

(a) - δ1,65-2.2 (4 H,m)
(b) - δ3.55 (4 H,t)
(c) - δ6.25 (1 H, d,J = 16 Hz)
(d) - δ5.96 (2 H,s)
(e) & (f)- δ6.65-7.65 (6 H)

OKOLASIN (R = $OCH_3$)

(a) - δ1.7 -2.05 (4 H,m)
(b) - δ3.58 (4 H,t)
(c) - δ6.25 (1 H, d, J = 16 Hz)
(d) - δ5.96 (2 H, s)
(e) - δ6.45 - 7.60 (5 H)
(f) - δ3.81 (3 H,s)

The proposed structure I (R = $OCH_3$) was further confirmed by synthesis starting from 6-methoxy piperic acid. Pyrrolidine was taken in dry benzene and added to the acid chloride from 6-methoxy piperic acid. This on refluxing and chromatography over neutral alumina afforded a compound identical with the natural product (TLC, IR, m.p., m.m.p., IR, UV and NMR).

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