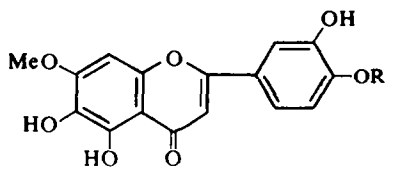
pedalitin (2), which it has been previously reported only as glycoside [4, 5].

The new flavone isolated from a $CHCl_3$ extract has been assigned structure (1) because it gives a positive Sr^{2+}–NH_3 test [6] for 5,6-dihydroxylation and shifts in the UV spectrum are consistent with this oxygenation pattern. A bathochromic shift with NaOMe with decrease in intensity indicates that the 4'-hydroxyl group is substituted. The absence of shift with either NaOMe or $NaOAc/H_3BO_3$ preclude the presence of 7-hydroxyl group or *o*-dihydroxyl group [7]. A shift in band I with $AlCl_3/HCl$ of about 20 nm indicates the presence of a hydroxyl function at the 6 position [8]. The NMR spectrum of (1) in DMSO-d_6 showed signals at δ 4.0 corresponding to two methoxyl groups, singlets at δ 6.7 (H-3), δ 6.9 (H-8) and δ 7.5 (H-2',6') and a doublet at δ 7.1 (H-5'). Peaks in the MS spectrum at 330(M^+), 315 (M-17) and 285 *m/e* (M-49) are in agreement with this structure. After methylation with Me_2SO_4 both (1) and (2) afforded 5-hydroxy-6,7,3',4'-tetramethoxyflavone (mp, UV, NMR) [4, 5].

The Et_2O extract (see Experimental) yielded pedalitin (mp, UV, NMR).



(1) R = Me, 5,6,3'-trihydroxy-7, 4'-dimethoxyflavone

(2) R = H, pedalitin

EXPERIMENTAL

Eupatorium inulaefolium was collected at Colonia Benítez, Province of Chaco, Argentina, February 1976 and a voucher specimen is deposited in the University Herbarium (Museo de Botanica, Universidad de Buenos Aires, Argentina). Air dried ground material (900 g) was extracted (24 hr) at room temp. with aq. MeOH. The aq. MeOH were evapd to dryness, redissolved in hot H_2O and partitioned with petrol, $CHCl_3$ and Et_2O. The petrol extract contained no flavonoids and was discarded. The $CHCl_3$ extract was evapd to dryness and passed twice through a column packed with Sephadex LH_{20} and eluted with C_6H_6, $CHCl_3$ and MeOH. The $CHCl_3$–MeOH eluates afforded 5,6,3'-trihydroxy-7,4'-dimethoxyflavone which crystallized from MeOH as yellow crystals (mp 245–247°). The Et_2O extract was applied to a polyamide column and upon elution with H_2O–MeOH (7:3) afforded 5,6,3',4'-tetrahydroxy-7-methoxyflavone which crystallized from MeOH (mp 295–297°) (lit. 300–301°) [4].

5,6,3'-Trihydroxy-7,4'-dimethoxyflavone. Purple (UV) to yellow-brown (UV/NH_3); R_fs: TBA 0.7, 15% HOAc = 0.02: UV λ_{max} (nm): MeOH, 232*sh*, 253*sh*, 285, 340; NaOMe, 260, 320*sh*, 367; $AlCl_3$, 240*sh*, 262*sh*, 302, 372; $AlCl_3$/HCl, 242*sh*, 257*sh*, 302, 367; NaOAc, 235, 290, 337; $NaOAc/H_3BO_3$, 235, 290, 337. NMR (60 MHz), (DMSO-d_6) using TMS as internal standard, signals at δ 7.5 (2H, *d*, J = 4 Hz), δ 7.1 (1H, *d*, J = 9 Hz), δ 6.9 (1H, *s*), δ 6.7 (1H, *s*) and δ 4.0 (6H, 2Me). MS, principal peaks at 330 (8%) (M^+), 312 (2.4%) ($M^+ - 17$), 283 (3.5%) ($M^+ - 49$), 268 (3.1%) ($M^+ - 64$) and 207 *m/e* (13%) ($M^+ - 125$). The IR and NMR spectra were identical to those of the synthetic compound, kindly provided to us by Prof. H. Wagner.

Methylation with Me_2SO_4 [4] afforded 5-hydroxy-6,7,3',4'-tetramethoxyflanone, yellow crystals from aq. MeOH (mp 188–189°) (lit. 189–190°) [4]. UV λ_{max} (nm), MeOH, 242, 275, 339 [5].

5,6,3',4'-tetrahydroxy-7-methoxy flavone (pedalitin). Purple (UV) to yellow-brown (UV/NH_3); R_fs TBA = 0.66, 15% HOAc = 0.02. Positive test with Sr^{2+}/NH_3. UV λ_{max} (nm): MeOH, 245*sh*, 285, 345; NaOMe, 264, 385; $AlCl_3$ 270, 300, 420; $AlCl_3$/HCl, 257*sh*, 295, 370; NaOAc, 260*sh*, 290, 360; $NaOAc/H_3BO_3$, 260*sh*, 290, 360. NMR (60 MHz) (DMSO-d_6) using TMS as internal standard, signals at δ 7.45 (2H, *d*, J = 3 Hz), δ 6.95 (1H, *d*, J = 9 Hz) δ 6.85 (1H, *s*), δ 6.65 (1H, *s*) and δ3.9 (3H, 1Me). Methylation with Me_2SO_4 afforded 5-hydroxy-6,7,3',4'-tetramethoxyflavone.

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ISOLATION OF STRICTOSIDINE (ISOVINCOSIDE) LACTAM FROM *NAUCLEA LATIFOLIA*

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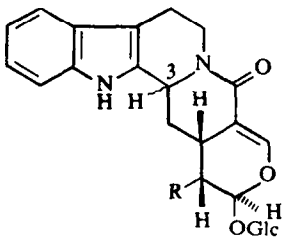
Key Word Index—*Nauclea latifolia*; Rubiaceae; indole alkaloid glycoside; strictosamide; artifact production.

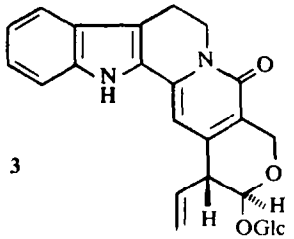
Nauclea latifolia heartwood, collected in the environs of Ahmadu Bello University, Zaria, Nigeria, was macerated and extracted with methanol. The orange residue left after removal of the solvent, was further

purified by a simple counter-current distribution. After three transfers an organic concentrate of indolic material was obtained, which was washed with pH 9 buffer to remove the phenolic cinnamates. Removal of the solvent gave an amorphous yellow powder in 1–2% yield which was identical in every respect with an authentic sample of strictosidine lactam [1] (**1a**). This was further characterized as its tetra-acetate. Similar treatment of the bark of *Nauclea latifolia* gave large amounts of strictosidine lactam together with traces of a compound with very similar spectra (R_f (1:1:1) 0.71) which was presumably an isomer.

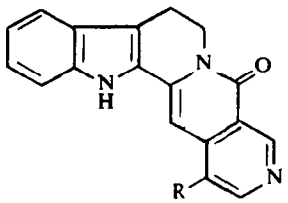
Previous workers had reported the presence in this plant of small quantities of alkaloids with structure (**2a–d**) [2]. In view of the use of ammonia in the extraction procedure and the ease with which dihydrovincoside lactam (**1b**) is converted into dihydroangustine (**2e**)



1 (a) 3α, R = CH = CH_2
(b) 3β, R = Et



3



2 (a) R = CH = CH_2
(b) R = CHOH.Me
(c) R = H
(d) R = Ac
(e) R = Et

[3], the possibility that the alkaloids (**2a–d**) are artifacts produced from strictosidine lactam during extraction, must be considered [4]. In this connection it was found that strictosidine lactam was slowly converted to a pyridone (**3**) on standing in solution, confirming the ease with which this compound undergoes aerial oxidation [5].

EXPERIMENTAL

Nauclea latifolia heartwood (50 g) was macerated and extracted with MeOH (5 × 200 ml). Removal of the solvent left an orange residue. This was subjected to a counter-current distribution between EtOAc and H_2O (500 ml fractions of each). After three transfers TLC examination showed aq. fractions 1 and 2 and organic fractions 2 and 3 to be essentially one component. The aq. fractions were concd and extracted with EtOAc (7 × 200 ml) and the combined extracts washed with pH 9 buffer (0.025 M Na tetraborate–boric acid) (200 ml). Removal of the solvent gave a yellow solid (0.61 g 1.2%) identical with strictosidine lactam. [TLC (toluene–EtOAc–MeOH, 1:1:1) R_f 0.67; $[\alpha]_D^{20}$ − 77° (MeOH, *c* 0.41), UV $\lambda_{max}^{(MeOH)}$: 290 (3.81), 230 (4.46), nm]. Acetylation (Ac_2O–Py gave an orange solid identical in every respect with strictosidine lactam tetra-acetate: TLC (ethyl acetate) R_f 0.7; $[\alpha]_D^{20}$ − 71° ($CHCl_3$ *c* 0.31), UV λ_{max}^{MeOH}: 290, 229 nm, CD $[\theta]_{265}^{MeOH}$: +2.2 × 10^4 degree cm^2/decimole, IR $\nu_{max}^{CHCl_3}$ 3470, 3270, 1760, 1665, 1650 cm^{-1}, NMR ($CDCl_3$) 100 MHz: 1.60 (1H, *s*, NH), 2.50–3.05 (5H, *m*, Aromatic H, C 17-H), 7.96, 8.04, 8.15 and 8.92 (12H, *s*, O.CO.Me), MS *m/e*; M^+ 666.2427 (71) (calculated for $C_{34}H_{38}N_2O_{12}$, 666.2425), 378 (7), 335 (12), 331 (16), 319 (16), 289 (7), 265 (16), 236 (5), 235 (7), 169 (100), 144 (14), 143 (19), 127 (21), 115 (9), 109 (57).

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TEINEMINE AND ISOTEINEMINE, TWO NEW ALKALOIDS FROM *VERATRUM GRANDIFLORUM*

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Key Word Index—*Veratrum grandiflorum*; Liliaceae; alkaloids; teinemine; (22*R*, 25*S*)-22,26-epiminocholest-5-ene-3β, 16α-diol; isoteinemine.

The isolation of solanidine [1] and etioline [2] as the main alkaloids from the terrestrial parts of budding *Veratrum grandiflorum* has already been reported. From the same plant material, in addition to veratramine,