EXPERIMENTAL

Isolation of lorostemin. The ground wood (7·5 kg) of *Lorostemon coelhoi* was extracted with benzene. The concentrated benzene solution gave a crystalline mass (80 g) which was separated by filtration from an oil (5 g). The crystals plus some ahering oil were recrystallized repeatedly from hot acetone, yielding in the first crops the photodimer. The mother liquors were evaporated. The residue was freed from oil by washings with light petrol. and recrystallized from hot MeOH giving lorostemin (I). The presence of lorostemin in the wood of *Lorostemon negrensis* was demonstrated by preparative TLC.

Lorostemin (I). Yellow crystals, m.p. 258–259° (Me_2CO) (Found: C, 67·15; H, 4·80. $C_{19}H_{16}O_6$ requires: C, 67·05; H, 4·75%). λ_{max}^{EtOH} (nm): 275, 338 (ε 42 000, 19 000); no $AlCl_3$/HCl shift; $\lambda_{max}^{EtOH+AlCl_3}$ (nm): 270, 350 (ε 34 500, 17 000); $\lambda_{max}^{EtOH+NaOAc+H_3BO_3}$ (nm): 270, 347 (ε 37 000, 19 400); $\lambda_{max}^{EtOH+NaOAc}$ (nm): 263, 370 (ε 38 500, 29 600); $\lambda_{max}^{EtOH+NaOH}$ (nm): 268, 372 (ε 39 000, 25 000). Gibbs test:[4] negative. ν_{max}^{KBr} (cm^{-1}): 3500, 1645, 1610, 1585, 1470, 1370, 1290, 1220, 1190, 1130, 1085, 960, 860, 840, 780, 740, 730. PMR [$(CD_3)_2CO$, τ]: 2·06 (*s*, H-8), 2·33 (*s*, H-5), 3·35 (*s*, H-2), 3·17 (*d*, *J* 10·3 Hz, H-4′), 4·10 (*d*, *J* 10·3, H-5′), 6·00 (*s*, $OC\underline{H}_3$), 8·41 (*s*, two $C\underline{H}_3$).

Di-O-methyllorostemin was obtained with CH_2N_2 in Et_2O and washings of the reaction product with 3% aq. NaOH, as light yellow crystals, m.p. > 280°. ν_{max}^{KBr} (cm^{-1}): 1638, 1600, 1560, 1500, 1460, 1448, 1428, 1381, 1370, 1270, 1245, 1223, 1205, 1175, 1158, 1116, 1110, 1090, 1015, 985, 890, 870, 830, 820, 785, 770, 725, 690. PMR ($CDCl_3$, τ): 2·38 (*s*, H-8), 3·20 (*s*, H-5), 3·43 (*s*, H-2), 3·28 (*d*, *J* 10·0 Hz, H-4′), 4·32 (*d*, *J* 10·0 Hz, H-5′), 6·05 (*s*, three $OC\underline{H}_3$), 8·53 [*s*, $C(C\underline{H}_3)_2$].

Di-O-acetyllorostemin was obtained as crystals, m.p. 195–196° (light petrol.–C_6H_6). ν_{max}^{KBr} (cm^{-1}): 1765, 1658, 1625, 1603, 1562, 1490, 1465, 1445, 1376, 1280, 1180–1210, 1157, 1124, 1105, 1020, 933, 890, 840, 780. PMR ($CDCl_3$, τ): 1·98 (*s*, H-8), 2·72 (*s*, H-5), 3·43 (*s*, H-2), 3·20 (*s*, *J* 10·0 Hz, H-4′), 4·32 (*s*, *J* 10·0 Hz, H-5′), 6·11 (*s*, $OC\underline{H}_3$), 7·70 (*s*, two $COC\underline{H}_3$), 8·53 [*s*, $C(C\underline{H}_3)_2$]. MS. M 424 (100%), *m/e* (%) 409 (97), 382 (20), 368 (88), 340 (24), 339 (15), 325 (71), 324 (18), 312 (5), 311 (15), 310 (15), 297 (10), 296 (12), 295 (15), 282 (5), 281 (9), 153 (5), 115 (6). *De-O-methyllorostemin* was obtained by treatment of I with conc. H_2SO_4 at room temp. (5 min.), addition of the mixture to crushed ice and filtration. Intensely greenish-yellow crystals, m.p. 182–184°. Gibbs test:[4] negative.

Lorostemin photodimer. Red crystals, m.p. 272–274° (Me_2CO) (Found: C, 67·21; H, 4·81. $C_{38}H_{32}O_{12}$ requires: C, 67·05; H, 4·75%). ν_{max}^{KBr} (cm^{-1}): 3500, 1645, 1590, 1560, 1530, 1465, 1455, 1376, 1322, 1290, 1265, 1250, 1200, 1170, 1120, 1005, 1090, 970, 850, 845, 840, 782, 740, 735, 700. MS: *m/e* (%) 340 (44), 339 (16), 325 (100), 312 (4), 311 (16), 310 (9), 297 (6), 296 (9), 295 (9), 282 (5), 281 (5), 162·5 (8), 153 (7), 115 (7). The *acetate* was obtained as white crystals, m.p. 188–190° (light petrol.–C_6H_6). PMR ($CDCl_3$, τ): 1·87 (*s*, H-8), 2·65 (*s*, H-5), 3·36 (*s*, H-2), 5·35 (*d*, *J* 6·0 Hz, H-4′), 5·85 (*s*, $OC\underline{H}_3$), 7·28 (*d*, *J* 6·0 Hz, H-5′), 7·60 (*s*, $COC\underline{H}_3$), 8·45 (*s*, low intensity peak due to $CC\underline{H}_3$ of a secondary product), 8·68 (*idem.*), 8·77 (*s*, two $CC\underline{H}_3$), 9·12 (*s*, two $CC\underline{H}_3$).

Acknowledgement—The authors are indebted to Prof. E. Wenkert, Indiana University, Bloomington, Indiana, U.S.A., for the MS.

Phytochemistry, 1973, Vol. 12, pp. 948 to 949. Pergamon Press. Printed in England.

THALICMININE FROM *OCOTEA PUBERULA*

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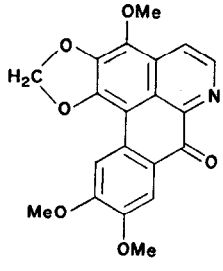
(*Received* 10 *November* 1972. *Accpeted* 5 *December* 1972)

Key Word Index—*Ocotea puberula*; Lauraceae; thalicimine.

Plant. Ocotea pubercula (Nees et Mart) Nees from Provincia de Misiones (Argentina).

Previous work. Ocoteine,[1,2] dehydroocoteine,[3] and a compound reported as a 9,10-phenanthraquinone[1,2] were previously isolated. Didehydroocoteine[3] was identified as a component of the extracts. *Previous work.* On other species, reported ocoteine from *Nectandra saligna* (Nees et Mart) Nees, *Phoebe porfiria* (Gris) Mez,[4] and *Thalictrum fendleri* Engelm et A. Gray.[5] Ocoteine and thalicminine (I) together with other alkaloids from *T. isopyroides* C. A. Mey,[6] and T. *minus* L.,[7] thalicminine from T. *simplex* L.[8]

Present work. The plant material was extracted following previous procedures,[2] and the $CHCl_3$ solution after concentration yielded (0·25%) of the product reported as a quinone[1,2] m.p. 275–277° (d), It was identified as thalicminine (I) (lit[9] m.p. 274–275°, lit.[7] 263–265°). (Found: C, 65·3; H, 4·56, N, 3·58; $C_{20}H_{15}NO_6$ (365·33) required: C, 65·80; H, 4·10; N, 3·83%) UV (EtOH) λ_{max} (log ϵ): 254 (4·70); 280 (4·53); 357 (4·43); 452 (3·4).[7] IR (KBr), 1636 cm^{-1} (CO) 1270, 1140 (OMe) 965 (OCH_2O).[7] NMR CF_3COOH), 4·20 (3H, *s*, $OC\underline{H}_3$), 4·25 (3H, *s*, $OC\underline{H}_3$), 4·55 (3H, *s*, $OC\underline{H}_3$), 6·65 2H, *s*, $OC\underline{H}_2O$), 8·10 (1H, *s*, Ar$\underline{H}$ C-4), 8·35 (1H, *s*, Ar$\underline{H}$ C-8), 8·88 (1H, *s*, Ar$\underline{H}$ C-5) and 8·94 (1H, *s*, Ar$\underline{H}$ C-11).[9] MS (direct inlet), M^+ 365, and peaks at *m/e* 350 (M–Me), 322 (M–Me–CO), 335, 320 (M–Me–CH_2O). 307 (M–Me–CO), 292, 290, 223, 219, 210, 205, 182·5 (M^{2+}).[10]



(I)

Thalicminine (I) could be obtained by oxidation of ocoteine with potassium permanganate in acetone,[1,7] and dehydroocoteine was identified as an intermediate product. Thalicminine (I) was also prepared with 10% yield by oxidation of the latter following a similar procedure.

Acknowledgements—We thank Dr. Daniel Banes (FDA, Washington, U.S.A.) for the MS and to Dr. José Amorín (INFyB) for collection of the plant material.

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