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SHORT COMMUNICATION

ISOLATION OF 5-HYDROXY-3,6,7,3′,4′-PENTAMETHOXY FLAVONE FROM *VITEX NEGUNDO*

A. BANERJI, M. S. CHADHA and V. G. MALSHET

Biology Division, Bhabha Atomic Research Centre, Trombay, Bombay-74

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Abstract—5-Hydroxy-3,6,7,3′,4′-pentamethoxyflavone has been isolated from the leaves of *Vitex negundo*.

PLANTS belonging to the genus *Vitex* (Verbenaceae) are said to possess hormone-like properties.[1] An insect moulting hormone, 20-hydroxyecdysone, has recently been isolated from *Vitex megapotamica*.[2] The leaves of *V. negundo* have been studied by us but no such activity was observed. The present note describes the isolation and characterization of 5-hydroxy-3,6,7,3′,4′-pentamethoxyflavone which is of rare occurrence.[3]

The plant was collected during May–June from the campus of the Research Centre where it grows wild. The petroleum ether extract of the leaves, on concentration, deposited an amorphous mass which on trituration with ether left a crystalline solid. This, on crystallization from acetone–methanol, furnished thick rectangular crystals (m.p. 163°) analysing for $C_{20}H_{20}O_8$ (yield, 0·002 per cent of dried leaves). I.r. spectrum of the compound shows hydroxyl (2·85 μ), conjugated carbonyl (6·0 μ) and aromatic (6·10, 6·30, 6·42, 6·60 μ) absorptions. U.v. spectrum ($\lambda_{max}^{CH_3OH}$, 254, 273 sh. and 348 nm; log ϵ, 4·27, 4·25 and 4·35) suggests a flavone skeleton for the compound. It gives characteristic colour tests for 3-substituted flavones.[4] In the NMR spectrum, signals at 6·97δ (1 H, doublet, J=9 cps) and 7·75δ (2 H, multiplet) suggest a 3′,4′-disubstituted B-ring[5] and this is supported by the presence of a shoulder at 273 nm in the u.v. spectrum.[6] A singlet (1 H) at 6·48δ can be assigned to an uncoupled proton in the ring A. A low field one-proton singlet at 12·57δ, which disappeared on D_2O exchange, can account for the 5-hydroxyl group while no other signal was affected by the exchange. The presence of a chelated 5-hydroxyl group was further confirmed by (*a*) a positive ferric test, (*b*) a bathochromic shift in the u.v. absorption maxima to 262, 282 and 362 nm on the addition of $AlCl_3$ and (*c*) a shift to the higher wave-length of the carbonyl absorption on acetylation and methylation to 6·10 and 6·12 μ respectively, in the i.r. spectrum.[7]

[1] M. SIRAIT, H. RIMPLER and R. HÄNSEL, *Experientia* **18**, 72 (1962).
[2] H. RIMPLER and G. SCHULZ, *Tetrahedron Letters* 2033 (1967).
[3] J. B. HARBORNE, *Comparative Biochemistry of the Flavonoids*, pp. 57, 228, Academic Press, London (1967).
[4] K. VENKATARAMAN, in *Progress in the Chemistry of Organic Natural Products* (edited by L. ZECHMEISTER), Vol. 17, p. 1 (1959).
[5] T. J. MABRY, J. KAGAN and H. RÖSLER, *Phytochem.* **4**, 177 (1965).
[6] L. JURD, in *The Chemistry of Flavonoid Compounds* (edited by T. A. GEISSMAN), p. 107, Pergamon Press, Oxford (1962).
[7] S. BALAKRISHNA, J. D. RAMANATHAN, T. R. SESHADRI and B. VENKATARAMANI, *Proc. R. Soc.* **268A**, 1 (1962).

The singlets at 3·95, 3·91 and 3·85δ integrating to fifteen protons suggest the presence of five methoxyl groups. On prolonged refluxing with dimethyl sulphate in acetone in the presence of K_2CO_3, 3,5,6,7,3′,4′-hexamethoxyflavone, m.p. 140° (hexamethylquercetagetin), was obtained. On the basis of the above evidence, the parent compound is assigned the structure: 5-hydroxy-3,6,7,3′,4′-pentamethoxyflavone. For obtaining an authentic sample, quercetagetin, extracted from the *Tagetes erecta* flowers,[8] was partially methylated (5·1 mol. $(CH_3)_2SO_4$; 4 hr) to yield 5-hydroxy-3,6,7,3′,4′-pentamethoxyflavone and the two samples were indistinguishable (m.p., mxd. m.p., i.r., TLC). The acetates (m.p. 161°) of these samples were also found to be identical in all respects.

[8] P. S. RAO and T. R. SESHADRI, *Proc. Indian Acad. Sci.* **14A**, 289 (1941).