

SHORT COMMUNICATION

ISOLATION AND IDENTIFICATION OF XANTHYLETIN IN CITRUS ROOTS

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Plants. *Citrus sinensis* (L.) Osbeck (Shamouti orange), *C. aurantium* L. (sour orange), *C. limettioides* (Tan.) (Palestine sweet lime), *C. paradisi* Macfadyen (Marsh seedless grape fruit) and *C. limon* L. Burm. f. (Eureka lemon). All belong to the Rutaceae.

Previous work. Bark, wood and roots of Rutaceae.¹⁻⁴

Roots. Washed and dried material of 6-30-month-old seedlings was ground to pass 40-mesh screen and extracted in a soxhlet (40 g, 200 ml of 40-60° b.p. petrol ether, 8-10 hr). After concentration the extract was chromatographed on Silica gel-H (Merck) column (40 × 2.5 cm) prepared with cyclohexane. Elution started with 1% EtOAc in cyclohexane. The concentration of EtOAc was gradually increased to 2, 5, 7 and 15%, the eluate being monitored by TLC. A compound having bright-blue fluorescence in u.v., with R_f 0.34 [TLC successive development with EtOAc-cyclohexane (1:9) and (2:8)] was obtained. Fractions containing this compound were combined, dried, evaporated and recrystallized from petrol ether-EtOAc. It was identified as xanthyletin ($C_{14}H_{12}O_3$) based on NMR, mass spectroscopy ($m/e = 228$), u.v. (λ_{max} methanol-223 nm ($\log \epsilon$ 4.22), 264 nm ($\log \epsilon$ 4.24), (301 nm) ($\log \epsilon$ 3.70), (314 nm) ($\log \epsilon$ 3.77) and 345 nm ($\log \epsilon$ 4.09), i.r. and m.p. (130-131°). NMR, u.v. and $\log \epsilon$ values were superimposable with those published by Reich *et al.*¹ with the addition of the 223 nm peak reported by us. Mixed m.p. with an authentic sample was undepressed.

Stem. Xanthyletin was also detected in the stem of young sour orange seedlings.

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¹ J. VON REISCH, K. SZENDREI, E. MINKER and I. NOVAK, *Planta Med.* 17, 116 (1969).

² F. E. KING, J. R. HOUSLEY and T. J. KING, *J. Chem. Soc.* 1392 (1964).

³ F. M. DEAN, *Progress in the Chemistry of Organic Natural Products* (edited by L. ZECHMEISTER), Vol. IX, p. 225, Springer-Verlag, Vienna (1952).

⁴ J. C. BELL, A. ROBERTSON and T. S. SUBRAMANIAN, *J. Chem. Soc.* 627 (1936).