

HYMENOXIN A FLAVONE FROM
*HELIANTHUS ANGUSTIFOLIUS**

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A chloroform extract of *Helianthus angustifolius* L (Tribe Heliantheae), collected in East Tennessee, yielded after chromatography a polar flavone whose m.p and UV properties were nearly identical to those of hymenoxin (5,7-dihydroxy-6,8-3',4'-tetramethoxyflavone, I) ¹ This compound, a member of the uncommon group of fully oxygenated A-ring flavones, ² was first isolated from *Hymenoxys scaposa* (Compositae, Helenieae). ³

The NMR and MS of the *Helianthus* flavone in conjunction with its UV spectra were indicative of structure I, ^{1,4} and a direct comparison with authentic hymenoxin (m m p, TLC, UV) established its identity The co-occurrence of hymenoxin in the tribes Helenieae and Heliantheae is consistent with the close relationship of these groups pointed out by Strother ⁵

EXPERIMENTAL

Isolation of hymenoxin from Helianthus angustifolius L The dried and ground plant material (2.85 kg) of *Helianthus angustifolius* L (Voucher No HA-TC971-TW) ⁶ collected in September 1971 near Tracy City, Tennessee was exhaustively extracted with CHCl₃ at room temp The CHCl₃ extract was evaporated to dryness *in vacuo* and the crude syrup taken up in 800 ml of hot H₂O-EtOH (3:1) The mixture was stirred and heated for 15 min and cooled to room temp The aqueous extract was filtered through Celite and extracted repeatedly with 150 ml portions of CHCl₃ The combined organic phase was dried (MgSO₄), filtered and evaporated to dryness Repetitions of this procedure yielded 16 g of a combined final extract The thick oil was chromatographed on silica gel, eluting with C₆H₆ followed by increasing proportions of CHCl₃ Fractions were grouped according to TLC and rechromatography of combined later fractions yielded 15 mg of a greenish powder Recrystallizations from MeOH gave pure hymenoxin (I) (9 mg, m p 213–215°), identified by direct comparison with an authentic sample (m m p, TLC, UV) ⁷ The MS of I displayed fragment ions at *m/e* 374, 359, 341, 331, 197 and 169 NMR (CDCl₃) 4.00 ppm (4 × 3H, s), 6.57 (1H, s), 6.99 (1H, d, *J* 9 Hz), 7.58 (1H, dd, *J* 9.2 Hz), 7.40 (1H, d, *J* 2 Hz), 6.38 (1H, bs), 12.65 (1H, s)

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¹ MABRY, T J, MARKHAM, K R and THOMAS, M B (1970) *The Systematic Identification of Flavonoids*, Springer, New York

² HARBORNE, J B (1967) *Comparative Biochemistry of the Flavonoids*, pp 42–46, Academic Press, New York

³ THOMAS, M B and MABRY, T J (1967) *J Org Chem* **32**, 3254

⁴ KINGSTON, D G I (1971) *Tetrahedron* **27**, 2691

⁵ STROTHER, J L, private communication

⁶ Identified by Dr GENE S VAN HORN, Department of Biology, University of Tennessee at Chattanooga a voucher specimen has been deposited in the U T C Herbarium

⁷ An authentic sample was kindly supplied by Dr T J MABRY, University of Texas