

SHORT COMMUNICATION

ISOLATION OF 3,3',4',5,5',7,8-HEPTAMETHOXYFLAVONE FROM *MURRAYA EXOTICA**

B. S. JOSHI and V. N. KAMAT

CIBA Research Centre, Goregaon, Bombay 63, India

(Received 29 July 1969)

Abstract—3,3',4',5,5',7,8-Heptamethoxyflavone (hibiscetin heptamethyl ether) has been isolated from the leaves of *Murraya exotica* L.

Plant material. *Murraya exotica* L. (Rutaceae) collected at Thenmala Forest, Kerala, in October 1965.

Previous work. Chemical.¹⁻⁴

Leaves. Extracted with MeOH and the crude extract (12 g) chromatographed on (120 g) silica gel (0.05–0.2 mm).

Compound isolated. Elution with CHCl₃—2% MeOH gave pale yellow needles recrystallized from CH₂Cl₂—EtOH (130 mg), m.p. 190°, *R_f* 0.7 (TLC on Si gel; CHCl₃:3% MeOH). Analyses for C₂₂H₂₄O₉ (M⁺ at *m/e* 432). A red colour in Shinoda test and the u.v. spectrum $\lambda_{\max}$ EtOH 209, 252, 266 (infl.), 274, 310 (infl.) and 348 nm; log ϵ , 4.73, 4.19, 4.25, 4.27, 4.16 and 4.22 are characteristic of a flavonoid compound. In the NMR spectrum (solvent, CDCl₃), signals at 7.57 δ (2H, singlet), 6.45 δ (1H, singlet) and 4.0 δ (21H, multiplet) indicated it to be a heptamethoxyflavone. The singlets at 7.57 δ and 6.45 δ should be attributed to the 2',6'-protons of ring B¹ and the 6-proton of ring A.⁵ This data suggested 3,3',4',5,5',7,8-heptamethoxyflavone as the probable structure. Methylation of 5,7,8-trihydroxy-3,3',4',5'-tetramethoxyflavone (m.p. 255°) with dimethyl sulphate gave the completely methylated flavone (heptamethyl ether of hibiscetin), m.p. 190°, identical with natural compound (mxd. m.p., i.r., TLC).

Acknowledgements—The authors thank Professor T. R. Seshadri for a sample of 3,3',4',5'-*O*-tetramethyl ether of hibiscetin and Dr. T. R. Govindachari for his interest in the work.

* Communication No. 170 from CIBA Research Centre.

¹ B. S. JOSHI and V. N. KAMAT, *Indian J. Chem.* 7 (1969).

² D. L. DREYER, *J. Org. Chem.* 33, 3471 (1968).

³ D. P. CHAKRABORTY, B. K. CHOWDHURY and B. C. DAS, *Tetrahedron Letters* 3471 (1967).

⁴ W. KARRER, *Konstitution und Vorkommen der organischen Pflanzenstoffe*, Birkhäuser Verlag, Basel (1958).

⁵ C. A. HENRICK and P. R. JEFFERIES, *Austr. J. Chem.* 17, 934 (1964).