EXPERIMENTELLES

A. dracunculus (AR-913) wurde aus Achänen vom Botanischen Garten Krefeld (BRD) im Botanischen Garten der Universität Wien angezogen. Davon extrahierte man 150 g frische, zerkleinerte Wurzeln 2 Tage bei Zimmertemperatur mit Ether–Petrol (1:2). Der Extrakt wurde zunächst grob durch SC (Si gel, Akt. St. 2–3) und dann durch DC (Si gel, GF_{254}) getrennt. Als Laufmittel dienten Ether–Petrol (60–80°)-Gemische.

8-Hydroxycapillarin (**5**). Farblose Kristalle aus Ether, Schmp. 158°. IR ($CHCl_3$, Beckman IR 9) cm^{-1}: OH 3600–2700; C=O 1690; C=COR 1650; Aromat 1610, 1570. UV $\lambda_{max}^{Et_2O}$ nm: 338, 257, 235. MS (Varian MAT 711, 70 eV, Direkteinlaß): M^+ *m/e* 214.063 (100%) (ber. für $C_{13}H_{10}O_3$ 214.06); $-{}^{\cdot}Me$ 199 (5); $-CO$ 186 (11); $-{}^{\cdot}CHO$ 185 (7); $-{}^{\cdot}CH_2C{\equiv}CMe$ 161 (51); 161 $-CO$ 133 (11); 133 $-CO$ 105 (81). ^{1}H-NMR (Bruker WH 270, TMS als innerer Standard, $CDCl_3$): *t* 6.62 (4-H, J = 1); *dd* 6.95 (5-H, J = 8, 0.7); *dd* 7.59 (6-H, J = 8, 7.5); *dd* 6.88 (7-H, J = 7.5), 1.0); *dq* 3.45 (1′-H, J = 2.5, 1); *t* 1.91 (4′-H, J = 2.5); *s* 10.03 (OH).

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A NEW FLAVONE FROM *TERMINALIA ARJUNA* FRUITS

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Key Word Index—*Terminalia arjuna*; Combretaceae; 5,7,2′,4′-tetramethoxy flavone.

Earlier work [1] on the fruits of *Terminalia arjuna* W & A led to the isolation of sitosterol, arjunic acid, mannitol and tannins. A re-investigation of the fruits of *T. arjuna* has now yielded a new flavone from the ether-soluble portion of the alcohol extract of *T. arjuna* fruits.

Its colour reactions and UV data indicated it to be a flavone lacking free hydroxyl groups. Its NMR spectrum in $CDCl_3$ showed the presence of four methoxyl groups, in addition to other expected signals. On degradation with alcoholic KOH, it gave 2,4-dimethoxybenzoic acid and 2,4-di-*O*-methylphloroacetophenone along with a small amount of the corresponding diketone, 2,4-dimethoxy-benzoyl-(2-hydroxy-4,6-dimethoxybenzoyl)-methane indicating the structure as 5,7,2′,4′-tetramethoxy flavone. This was confirmed by its synthesis using the Baker–Venkataraman method.

EXPERIMENTAL

Extraction and isolation. Air-dried *T. arjuna* fruits (5 kg) were successively extracted with hot petrol and EtOH. The EtOH extract was partitioned between Et_2O-soluble and Et_2O-insoluble portions. Et_2O-soluble portion on column chromatography and subsequent PLC yielded the new flavone.

5,7,2′,4′-Tetramethoxy flavone crystallized as needles from alcohol (20 mg), mp 179–80°. R_f: 0.29 (TEF, 5:4:1); 0.25 (C_6H_6–MeCOMe, 8:2). (Found: C, 66.8; H, 5.4. $C_{19}H_{18}O_6$ requires: C, 66.7; H, 5.3%). λ_{max}^{MeOH} nm log (ε): 245 (4.34), 340 (4.51); ν_{max}^{KBr} cm^{-1}: 1630, 1425, 1340, 1280. NMR ($CDCl_3$): δ 3.88 (3H, *s*,—OMe), 3.91 (3H, *s*,—OMe), 3.94 (3H, *s*,—OMe), 3.97 (3H, *s*,—OMe), 6.36 (1H, *d*, J = 2.5 Hz, C-6 proton), 6.5 (1H, *s*, C-8 proton), 6.6 (2H, *q*, C-3′, 5′ protons), 6.95 (1H, *s*, C-3 proton), 7.82 (1H, *d*, J = 9.5 Hz, C-6′ proton).

Synthesis. A mixture of 2,4-di-*O*-methylphloroacetophenone (2 g), 2,4-dimethoxybenzoyl chloride (2.5 g) and C_5H_5N (20 ml) was heated at 100° for 3 hr. The resulting ester was purified by column chromatography and crystallized from EtOH (1.5 g), mp 89–90°. (Found: C, 63.6; H, 6.0. $C_{19}H_{20}O_7$ requires: C, 63.3; H, 5.6%). ν_{max}^{KBr} cm^{-1}: 1730, 1630, 1600, 1450 and 1410. 2-(2,4-Dimethoxybenzoyloxy)-4,6-dimethoxy acetophenone (1 g) in dry C_5H_5N (10 ml) was treated with powdered KOH (1 g) and the mixture shaken vigorously for 2 hr with occasional warming. The resulting 2,4-dimethoxybenzoyl-(2-hydroxy-4,6-dimethoxybenzoyl)-methane [2] crystallized from EtOH (800 mg), mp 120°; ν_{max}^{KBr} cm^{-1}: 3246, 1600, 1425. The diketone (500 mg) was gently refluxed with HOAc (5 ml) and fused NaOAc (1 g) in an oil bath for 3 hr. The resulting flavone [2, 3] crystallized from EtOH (300 mg), mp 179–80°. It was identical with the natural sample (mmp, co-TLC, UV and IR).

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