SPECIALIA

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The Synthesis of 5,7,4'-Trihydroxy-3,8,3'-Trimethoxyflavone

In 1967, a flavonoid pigment was isolated from *Cyanostegia angustifolia* Turcz. (Verbenaceae) by E.L. GHISALBERTI et al.¹ They established the pigment as 5,7,4'-trihydroxy-3,8,3'-trimethoxyflavone (I) on the basis of analytical, spectroscopic and degradative studies. The pigment, an isomer of jaceidin (5,7,4'-trihydroxy-3,6,3'-trimethoxyflavone) (II)², has 5,7,8-oxygenated ring A instead of 5,6,7-oxygen pattern of II. In a previous paper³, the authors reported the synthesis of II from 2,4,6-trihydroxy-3, ω -dimethoxyacetophenone (III). This paper will describe the synthesis of I confirming the proposed structure.

Condensation of the ketone (III) with *O*-benzylvanillic anhydride in the presence of triethylamine gave a compound (m.p. 206–208°; yield 9%) in addition to 4'-benzyloxy-5,7-dihydroxy-3,6,3'-trimethoxyflavone (IV) (m.p. 180–181.5°; yield 20%) which had been previously re-

ported³. The compound was assigned to be an isomer of IV, 4'-benzyloxy 5,7-dihydroxy-3,8,3'-trimethoxyflavone (V) (golden yellow prisms, IR 3100 (broad) (OH), 1653 cm⁻¹ (γ -pyrone) (Nujol), UV $\lambda_{\text{max}}^{\text{EtOH}}$ nm (log ϵ): 257 (4.23), 276 (4.29), 340 (4.18). Found: C, 66.91; H, 4.70. C₂₅H₂₂O₈ requires: C, 66.66; H, 4.92%). Then, catalytic debenzylation of V with hydrogen gave the desired flavone (I, m.p. 213.5–215°, IR 3480, 3280 (OH), 1653 cm⁻¹ (γ -pyrone) (Nujol), UV $\lambda_{\text{max}}^{\text{EtOH}}$ nm (log ϵ): 260 (4.23), 276.5 (4.29), 340 sh (4.17), 365 (4.21). Found: C, 59.79; H, 4.40. C₁₈H₁₆O₈ requires: C, 60.00; H, 4.48%) (the natural pigment⁴, m.p. 211–213° (lit.¹ m.p. 215–217°, UV $\lambda_{\text{max}}^{\text{EtOH}}$ nm (log ϵ): 260 sh (4.33), 277 (4.45), 332 (4.29), 365 (4.30)), whose identity with the natural pigment was confirmed by mixed melting point determination, IR- and UV-spectral comparison⁴. Its triacetate (VI) (m.p. 140–141°, after resolidifying, 147–148.5°) (the natural derivative⁵; m.p. 139–140°, after resolidifying m.p. 146–147°) (lit.¹ m.p. 143–145°) was prepared by a usual method.

Zusammenfassung. Die Synthese von 5,7,4'-Trihydroxy-3,8,3'-trimethoxyflavon aus 2,4,6-Trihydroxy-3, ω -dimethoxyacetophenon wird beschrieben. Das synthetische Präparat ist mit einem aus *Cyanostegia angustifolia* Turcz. isolierten Pigment identisch.

K. FUKUI, M. NAKAYAMA and T. HORIE

Department of Chemistry, Faculty of Science,
Hiroshima University,
and Department of Applied Chemistry,
Faculty of Engineering, University of
Tokushima (Japan), 1 February 1968.

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¹ E.L. GHISALBERTI, P.R. JEFFERIES and C.I. STACEY, Aust. J. Chem. 20, 1049 (1967).

² L. FARKAS, L. HÖRHAMMER, H. WAGNER, H. RÖSLER and R. GURNIAK, Chem. Ber. 97, 610 (1964).

³ K. FUKUI, T. MATSUMOTO, S. NAKAMURA, M. NAKAYAMA and T. HORIE, Experientia 24, 108 (1968).

⁴ The natural pigment, kindly supplied by Prof. P. R. JEFFERIES, the University of Western Australia, was measured in this laboratory.

⁵ This compound was prepared from the natural pigment⁴ in this laboratory.