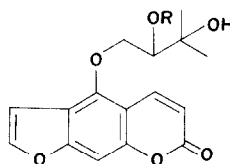


torin, imperatorin, oxypeucedanin, oxypeucedanin hydrate, peucenin, and a sterol mixture (MP, TLC, UV, IR, NMR, MS).

The concentrated methanolic extract was kept in a refrigerator. The precipitated hesperidin was filtered off and the fluorescing components of the mother liquor were separated on a polyamide column. Elution began with H_2O followed by H_2O -MeOH mixtures. TLC control of the fractions eluted with 20% MeOH showed the presence of several fluorescing polar compounds. They were separated by preparative TLC (silica gel) in $CHCl_3$ -MeOH 9:1. The most polar zones yielded a yellow (R_f : 0.22; system: $CHCl_3$ -MeOH 85:15), and three violet fluorescing compounds. One of the latter (R_f : 0.19) was identified (TLC, hydrolysis) as scopolin, the second (R_f : 0.25) as marmesinin. The third glycoside (R_f : 0.10) afforded on acidic hydrolysis the same aglycon as marmesinin. Its R_f -value suggests to be a diglycoside of marmesinin.

The yellow fluorescing glycoside crystallized

from MeOH in white needles, turning slowly yellow by heating (m. 210–220°, dec.). Its UV spectrum is typical of the 5-alkoxy-furocoumarin nucleus [2]; λ_{max} : 240 sh, 249, 259, 266, 308 nm; λ_{min} : 235, 255, 263, 275 nm. The glycoside was hydrolysed with $N H_2SO_4$, the aglycon extracted with EtOAc and identified as oxypeucedanin hydrate (1). The sugar moiety was identified by PC as D-glucose. Thus, the probable structure of the glycoside is 2.



(1) $R = H$

(2) $R = Glc$

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ISOLATION OF IPOLAMIIDE FROM *STACHYTARPHETA INDICA*

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Key Word Index—*Stachytarpheta indica*; Verbenaceae; iridoids; ipolamiide; hebenstreitia glycoside B.

Plant, *Stachytarpheta indica* VAHL. (Voucher specimen deposited at Faculty of Pharmaceutical Sciences, Chulalongkorn University, Department of Pharmacognosy). *Local name*, Ya Phanngu-Khieo, Pra-in-proei. *Source*, Ayudhaya, Thailand. *Previous work*, Occurrence of hebenstreitia glycoside B in *Stachytarpheta*-species [1].

Present work, Aerial parts (dry, 1200 g) of *Stachytarpheta* were macerated overnight with 80% EtOH. After decantation the residue was remacerated. The EtOH was removed under vacuum and the residual aqueous solution treated by standard procedures (lead acetate and charcoal method) [2].

Crystallization and recrystallization from EtOH afforded 14 g needles (1.17%). The properties of this compound and of its tetra- and penta-acetates are identical with those reported for ipolamiide [3] and its acetates respectively (analysis, m.p., optical rotation, UV, IR, NMR). M^+ of the penta-acetate m/e 616.

The compound is also identical (m.p., TLC, IR) with the hebenstreitia glycoside B isolated by Kooiman [1].

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of analyses and the measurement of the NMR and MS spectra. Thanks are due to Dr. P. Kooiman, Laboratorium for Algemeene en Technische Biologie, Technische Hogeschool, Delft, for the authentic sample of hebenstreitia glycoside B and to Prof. M. L. Scarpati, Instituto di Chimica Organica, Università di Roma, for the authentic sample of ipolamiide. Financial support by the Swiss National Science Foundation is gratefully acknowledged.

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A HYDROXY β -DIKETONE FROM LEAF WAX OF *FESTUCA OVINA**

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Key Word Index: *Festuca ovina*; Gramineae; leaf wax; 5-hydroxytritriacontane-12,14-dione.

Plant. *Festuca ovina* L. *Source.* Seeds of strain 1792 (glaucous strain) from Dr. R. P. Knowles, Canada Department of Agriculture, Saskatoon, Sask., (original seeds from Alma Ata, Kazakhstan, U.S.S.R.). *Previous work.* Tritriacontane-12,14-dione was isolated from leaf wax of *Festuca glauca* [1].

Present work. Wax was extracted from leaves (1.2% of dry wt) and hydroxy β -diketone (1.3% of wax) isolated as described previously [2]. The m.p. was 78° and $[\alpha]_D^{25} + 0.58^\circ$, $[\alpha]_{546}^{25} + 1.08^\circ$, $[\alpha]_{436}^{25} + 1.55^\circ$, $[\alpha]_{365}^{25} + 2.46^\circ$ (c. 2.2 in CHCl_3). GLC comparison with other hydroxy β -diketones [2] showed that it contained 33 carbons and that homologues were absent. The structure was shown to be 5-hydroxytritriacontane-12,14-dione by MS: *m/e* 508, 490 (M-H₂O), 472 (M-2H₂O), 451 (4.5 cleavage, 35% of base peak), 422 (5.6 cleavage + H), 365, 352, 337 (11,12-cleavage), 295 (13,14-cleavage) 276, 241, 238, 223 (14,15-cleavage-H₂O), 181 (12,13-cleavage-H₂O), 180, 100, 83, 81, 71, 69, 57, 55, 43 (base peak), 41. Peaks at 337 and 295 showed that the longer methylene chain was not hydroxylated and therefore the peak at 451 showed that the hydroxyl group was at C-5. Location of the hydroxyl group on a carbon separated from a terminal CH₃ group by 3 methylene groups was confirmed by the carbon-13 NMR spectrum (C-3 signal at 27.86 ppm; compare carbon-13 spec-

trum of 10-hydroxytetradecanoate [3]). Both spectroscopic methods indicated that isomers with the hydroxyl at other positions were absent.

This is the second hydroxy β -diketone to be isolated which occurs as a single isomer, the other being 25-hydroxyhentriacontane-14,16-dione from rye [2] and durum wheat [4]. 5-Hydroxyhentriacontane-14,16-dione has been found in wax from oats but 6-hydroxy and 7-hydroxy isomers were also present [3,5]. In the present compound the hydroxyl group is situated at carbon 8 with respect to the carbonyl group but in the other hydroxy β -diketones it is at carbons 6, 7, 9 or 10 which are often also the sites of double bond formation in fatty acids. There are, however, a number of hydroxy fatty acids with the hydroxyl group at carbon 8 [6].

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