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FLAVONES AND FLAVONE GLUCOSIDES FROM THE LEAVES OF *TRACHELOSPERMUM ASIATICUM*

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Key Word Index—*Trachelospermum asiaticum* var. *intermedium*; Apocynaceae; succinic acid; apigenin; luteolin; arctigenin; apigenin-7-glucoside; luteolin-7-glucoside; luteolin-4′-glucoside

Lignan glucosides were recently reported as the components of the stems of *Trachelospermum asiaticum* Nakai var. *intermedium* Nakai.[1,2]

We report the isolation of succinic acid (I) (0·0016% yield), apigenin (II) (0·0012% yield), luteolin (III) (0·0025% yield), arctigenin (IV) (0·0009% yield), apigenin-7-glucoside (V) (0·0006% yield), luteolin-7-glucoside (VI) (0·0011% yield) and luteolin-4′-glucoside (VII) (0·0026% yield) from the leaves.

The dried leaves were extracted with hot MeOH. The MeOH solution was evaporated, diluted with H_2O. The aqueous solution was extracted with Et_2O, $CHCl_3$ and AcOEt, respectively.

The AcOEt extract was chromatographed on silica gel column. I from eluate with $CHCl_3$, II, III and IV from eluate with $CHCl_3$–EtOH (1 : 19), V, VI and VII from eluate with $CHCl_3$–EtOH (3·17) were obtained in order. These compounds were identified with the authentic samples by IR, UV, TLC, PPC and m.m.p., respectively.

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NEW β-DIKETONES FROM *BUXUS SEMPERVIRENS*

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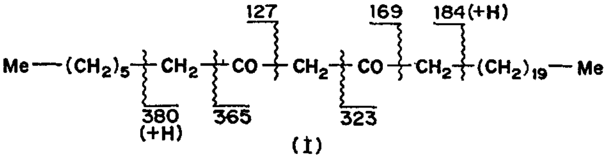
Plant. Buxus sempervirens L. *Source.* University campus of Louvain. A voucher specimen is deposited in the Herbarium of this University. *Previous work.* None.

Present work. Fresh leaves (550 g) were extracted by dipping in boiling light petrol. (30–45°) during 30 sec. Analytical TLC of the crude extract on silica gel G; C_6H_6; detection with Fast Blue B salt (Merck) revealed the presence of β-diketones (R_f 0·88). No trace of OH-β-diketones could be detected, although the spray reagent is very sensitive to compounds with a dicarbonyl group, as shown previously.[1] The waxy residue was adsorbed to basic Al_2O_3 and eluted with light petrol. (30–45°). This residue was chromatographed on a silica gel G column (10 × 2·2 cm) with light petrol.–C_6H_6 (9:1). Elution of the β-diketone zone with Et_2O gave 77 mg.

IR bands (KBr) ν_{max} 2900 (*s*), 2840 (*s*), 1715 (*m*), 1640 (*s*, β-diketone), 1465 (*s*), 1410 (*w*), some small peaks between 1300 and 1170, 1140 (*w*), 1100 (*w*), 790 (*m*), 780 (*m*), 730 (*m*) and 722 (*m*) cm^{-1}. UV band (Et_2O) λ_{max} 274 nm. From the characteristic IR band at 1640 cm^{-1}, the deep yellow colour with Fast Blue B salt,[1] the strong UV-absorption band at λ_{max} 274 nm and the identical chromatographic behaviour as compared with already known isomers we concluded that the analyzed compound was a long chain β-diketone.



$$Me—(CH_2)_5—CH_2—CO—CH_2—CO—CH_2—(CH_2)_{19}—Me$$

(I)

MS showed a molecular ion mass peak of 464 *m/e*, in agreement with the molecular formula $C_{31}H_{60}O_2$. Major ions were observed at 365, 127, 323 and 169 *m/e*, displaying the fragmentation shown on the structure (I). The peaks at 281, 211, 239 and 253 *m/e* on the other hand, which are characteristic for a *n*-C_{31}-14,16-dione,[2] were completely absent. McLafferty rearrangement accounted for the peaks at 380 and 184 *m/e*. Loss of water from the enol form gave peaks at 446 (M^+-18), 362, 347, 305, 166, 151 and 109 *m/e*. Since the MS revealed no indications for a branched alkyl chain, it could be concluded from the given data that the isolated compound has the structure of *n*-hentriacontane-8,10-dione (I). Weak corresponding MS-peaks were also found for the *n*-C_{29}- and *n*-C_{33}-dione. According to the relative intensity of these mass peaks, the diketone mixture was composed of 14% *n*-C_{29}-6,8-dione, 81% *n*-C_{31}-8,10-dione and 5% *n*-C_{33}-10,12-dione.

β-Diketones were first isolated from *Eucalyptus* by Horn *et al.*[3,4] and were afterwards cited several times, mainly from cereals.[5] In all these cases one of the two C=O functions was situated precisely in the centre of the molecule. Characteristic for the compounds presented here is the asymmetrical position of the dicarbonyl group, one of the alkyl chains being much longer than the other one.

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