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Phytochemical Studies at the Bulbs of Ornithogalum *Umbellatum* L.

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Phytochemical examination of the bulbs of *Ornithogalum umbellatum* L. led to the isolation a new steroidal compound. Its structure was determined to be 3-O-(2'-methoxy-4'-(2-pentenal))-phenyl sitosterol **(1)** by spectroscopic data and chemical evidence.

Key Words: Ornithogalum umbellatum, Liliaceae, bulbs, new aromatic compounds.

Introduction

Several cardenolide glycosides and several cholestane glycosides have been found in some species of *Ornithogalum* [1-8] in previous studies. In this study, we investigated the bulbs of O. u*mbellatum* L. phytochemically. The plant was collected from the Trakya region. $CHCl_3$ and $CHCl_3$:C_2H_5OH (2:1) extracts of the plant bulbs were separated and purified by chromatographic methods. The chemical structure of the four compounds isolated from the plant were elucidated by using UV, IR, NMR and FAB mass spectroscopy. One of them, 3-O-(2'-methoxy-4'-(2-pentenal))-phenyl sitosterol, is shown below.

Experimental

General. UV:Shimadzu UV 160 A visible spectrometer; IR: Shimadzu IR-470 spectrometer; ^{1}H NMR: 200 MHz (^{1}H) and 400 MHz (^{13}C) in $CDCl_3$; FAB mass: VG-zab spectrometer; TLC: Kieselgel 60 F 254 (Merck) precoated plates, spots were detected under a UV lamp or by heating after spraying with seric sulphate solution; CC: Kieselgel GF 254 (Fluka).

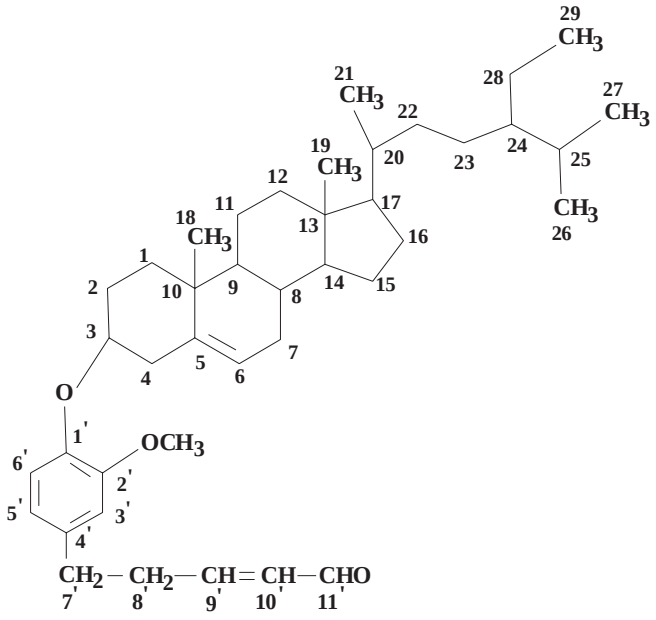
Plant material. *Ornithogalum umbellatum* L. was collected from Edirne in 1995 and identified by Assoc. Prof. G. Dalgıç; a voucher specimen is deposited in the Herbarium of the Biology Department, Faculty of Science-Arts, University of Trakya, EDTU 1923.

Extraction and Isolation of the Compounds. The bulbs of the plant were extracted with 70% EtOH in a Soxhlet apparatus, a month after their collection (4.5 kg). Extract was evaporated in an evaporator under vacuum. In order remove proteins, the saturated alcoholic solution of lead acetate was added to the condensed fraction and a precipitate was formed, and filtered [9]. The filtrate was extracted with

$CHCl_3$ followed by $CHCl_3$:EtOH (2:1) respectively. Two organic phases were condensed to dryness in vacuo. The residues obtained were 49 g and 0.95 g respectively. The $CHCl_3$ extract was subjected to CC. The column was eluted with $CHCl_3$:MeOH:H_2O (80:10:1), $CHCl_3$:MeOH:H_2O (75:16:2.2), $CHCl_3$:MeOH:H_2O (70:22:3.5), EtOAc:MeOH (95:5), and a gradient of EtOAc was added up to 100% followed by MeOH. The following compound was isolated.

***3-O-(2'-methoxy- 4'-(1-pentenal))-phenyl sitosterol* (1)**. (2.7 mg). UV λ_{max}($CHCl_3$) nm: 241, 274, 305; IR γ_{max}(Nujol) cm^{-1}: 2928, 1664, 1460-1625, 1148, 851, 812; ^{1}H NMR ($CDCl_3$): given in discussion; ^{13}C NMR ($CDCl_3$): δ 193.30 (C-11'), 143.30, 138.00, 132.70 132.50, 130.60, 130.40, 130.00 and 124.20 (C-1', C-2', C-3', C-4', C-5', C-6', C-9' and C-10'), 116.90 (C-5 or C-6), 111.30 (C-6 or C-5), 79.70 (C-3), 58.70 (OMe), 52.70, 48.40, 44.80, 42.30, 39.80, 34.40, 34.20, 32.20, 32.10, 31.90, 31.65, 31.60, 31.50, 30.80, 29.75, 29.70, 26.80, 23.60, 22.30, 21.90, 21.30, 16.65, 16.60, 14.50 and 14.40 were carbons of -CH_3, -CH_2, -CH at steroid ring and C-7', C-8'. The signals of two quaternary carbons in the steroid ring could not be observed because the signals of these carbons were very small; FAB mass m/z (rel. int.); 397 (100), 382 (15), 256 (10), 207 (10), 159 (10).

Results and Discussion



(1)

The UV spectrum of compound **(1)** showed maximum absorptions at 241, 274 and 305 nm. The IR spectrum of **(1)** indicated an α,β-unsaturated carbonyl (1664 cm^{-1}) group, an ether (1148 cm^{-1}) group and a 1,2,4-tri substitue aromatic (812 , 851 and 1460-1625 cm^{-1}) system. The ^{1}H NMR spectrum of **(1)** correlated with the suggested structure: δ 0.61 (3H, s, H-18), 0.94 (3H, s, H-19), 0.75 (3H, d, J=7 Hz, H-21), 0.82 (6H, d, J=7 Hz, H-26, 27), 0.80 (3H, dd, J=3,6 Hz, H-29), 1.2-2 (m, methylene protons), 2.25 (2H, t, J=7 Hz, H-8'), 2.7 (2H, t, J=6 Hz, H-7'), 3.48 (1H, m, H-3), 3.9 (3H, s, OMe), 5.3 (2H, m, H-9', 10'), 6.05 (1H, br d, J=4 Hz, H-6), 6.97 (1H, d, J=3 Hz, H-3'), 7.34 (1H, dd, J=3,6 Hz, H-5'), 7.35 (1H, d, J=8 Hz, H-6'), 9.8 (1H, s, H-11').

The ^{13}C NMR spectrum of **(1)** supported the suggested structure (see Experimental). The principal peaks in the FAB mass spectrum of **(1)** are m/z 397[M-$C_{12}H_{13}O_3$]$^+$ (the separation aromatic group from the steroid), m/z 382[397-CH_3]$^+$ and m/z 256[397-$C_{10}H_{21}$]$^+$ (the separation side chain from the steroid). In accordance with the ^1H and ^{13}C NMR data, the molecular formula is estimated as $C_{41}H_{62}O_3$. All the above evidence indicated that **(1)** is a 3-O-(2'-methoxy-4'-(2-pentenal))-phenyl sitosterol.

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