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DIVERSIVITTATIN, A NEW PHENYLPROPANOID DERIVATIVE FROM THE ROOTS OF *Ferula diversivittata*

M. Iranshahi,[1*] S. T. Hosseini,[2] A. H. Sahebkar,[2]
S. Suleman Khan,[3] and V. U. Ahmad[3]

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A new phenylpropanoid derivative, diversivittatin, was isolated from the roots of Ferula diversivittata. *The structure of this compound was elucidated by extensive spectroscopic methods including 1D (^{1}H and ^{13}C) and 2D NMR experiments (^{1}H–^{1}H COSY, HMQC, HMBC, and NOESY) as well as high-resolution EI-MS.*

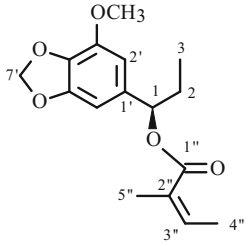
Keywords: *Ferula diversivittata*, Apiaceae, phenylpropanoid, diversivittatin, NMR.

The exclusively old world genus *Ferula* belongs to the family Apiaceae with about 130 species distributed throughout the Mediterranean area and Central Asia, specially in the former USSR and neighboring countries such as Iran. This genus is well documented as a good source of biologically active compounds such as sesquiterpene derivatives [1–6] and sulfur-containing compounds [7, 8].

The roots of *Ferula diversivittata* Regel & Schmalh.-Rech. have been used in folk medicine to prevent convulsion and hysteria [9]. The chemistry of the plant has previously been studied, and a few terpenoid coumarins have been identified to date [3]. Moreover, phenylpropanoids have also been isolated from some *Ferula* species such as *F. latipinna* [10], *F. pallida* [11], and *F. fukanensis* [12]. In the present work, we wish to report the isolation and the structure elucidation of a new phenylpropanoid from the roots of *F. diversivittata*, namely diversivittatin.

Normal-phase column chromatography of the dichloromethane extract of roots, followed by preparative TLC, afforded a new phenylpropanoid **1**, namely diversivittatin.

This compound was obtained as a yellow oil, and its molecular formula, $C_{16}H_{20}O_5$, was established by HR-EI-MS (*m/z* 292.1280, calcd 292.1311). The ^{1}H and ^{13}C NMR resonances (Table 1) of the compound were assigned by different 2D NMR experiments. The ^{1}H NMR spectrum showed resonances characteristic for two methyl singlets at δ 1.89 and 3.87, one olefinic resonance at δ 6.02 (1H, q, J = 7.2 Hz), and four methine resonances at δ 5.58 (1H, t, J = 6.8 Hz), 6.02 (1H, q, J = 7.2 Hz), 6.50 (1H, s) and 6.51 (1H, s). The latter two methines were ascribable to aromatic protons, suggesting the presence of a 1′,3′,4′,5′-substituted benzene ring, which was supported by the ^{13}C NMR spectrum.



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1) Department of Pharmacognosy, Biotechnology Research Center, School of Pharmacy, Mashhad University of Medical Sciences, Mashhad, Iran, fax: +98 511 8823251, e-mail: iranshahim@mums.ac.ir; 2) Department of Pharmacognosy, School of Pharmacy, Mashhad University of Medical Sciences, Mashhad, Iran; 3) University of Karachi, HEJ Research Institute of Chemistry, International Center for Chemical and Biological Sciences, Karachi 75270, Pakistan. Published in Khimiya Prirodnykh Soedinenii, No. 2, pp. 163–164, March–April, 2010. Original article submitted April 18, 2008.

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TABLE 1. ^{1}H NMR and ^{13}C NMR Data for Diversivittatin ($CDCl_3$, 500 MHz, δ, ppm, J/Hz)[a]

C atom	δ_H	δ_C	C atom	δ_H	δ_C
1	5.58 (t, J = 6.8)	77.1	6′	6.50 s	106.5
2	1.78 m	29.6	7′	5.93 s	101.4
3	0.83 (t, J = 7.2)	9.9	1″		167.2
1′	–	130.8	2″	–	128.0
2′	6.51 s	100.6	3″	6.02 (q, J = 7.2)	137.8
3′	–	143.3	4″	1.94 (d, J = 7.2)	15.7
4′	–	135.5	5″	1.89 s	20.6
5′	–	148.8	OCH_3	3.87 s	56.6

[a]Assignments were made by 2D COSY, HMQC, and HMBC data.

The ^{13}C NMR resonances showed 16 carbon signals, which could be resolved by DEPT and HMQC experiments into four methyls at δ_C 9.9 (C-3), 15.7 (C-4″), 20.6 (C-5″), and 56.6 (OCH_3), two methylenes at δ_C 29.6 (C-2) and 101.4 (C-7′), four methines at δ_C 77.1 (C-1), 100.6 (C-2′), 106.5 (C-6′), and 137.8 (C-3″), and six quaternary carbons at δ_C 128.0 (C-2″), 130.8 (C-1′), 135.5 (C-4′), 143.3 (C-3′), 148.8 (C-5′), and 167.2 (C-1″), including one carbonyl function (δ_C 167.2). In the HMBC spectrum, the correlations of H-1 (δ_H 5.58) with C-1′ (δ_C 130.8) and C-1″ (167.2); H-OCH_3 (δ_H 3.87) with C-3′ (δ_C 143.3); H-2′ (δ_H 6.51) with C-1′ (δ_C 130.8) and C-3′ (δ_C 143.3); H-6′ (δ_H 6.50) with C-1′ (δ_C 130.8) and C-5′ (148.8); H-7′ (δ_H 5.93) with C-4′ (δ_C 135.5) and C-5′ (148.8); H-3″ (δ_H 6.02) with C-1″ (δ_C 167.2); H-4″ (δ_H 1.94) with C-2″ (δ_C 128.0); and H-5″ (δ_H 1.89) with C-1″ (δ_C 167.2) confirmed the structure of the compound diversivittatin. The proposed structure was further supported by ^{1}H–^{1}H COSY data.

The configuration of the double bonds at C-2″ and C-3″ were determined as *Z* on the basis of the NOESY experiment, in which a cross-peak was observed from H-3″/H-5″ pairs, indicating an angeloyl moiety in the structure. Hence, the structure of the compound is as shown and named diversivittatin.

EXPERIMENTAL

General Experimental Procedures. UV spectra were obtained in CH_2Cl_2 on a Shimadzu UV-1650 PC spectrophotometer, and IR spectra were recorded on a Perkin–Elmer Paragin 1000 FT-IR spectrometer. ^{1}H and ^{13}C NMR, DEPT, ^{1}H–^{1}H COSY, HMQC, and HMBC spectra were recorded on Bruker Avance 500 and 600 MHz (with Cryo Probe), spectrometers for ^{1}H NMR, ^{13}C NMR, and 2D spectra.

The HR-EI-MS spectra was obtained on a JEOL JMS HX-110 mass spectrometer. Column chromatography was conducted with silica gel 230–400 mesh (Merck). Preparative TLC was performed on silica gel GF_{254s} plates (20 × 20 cm, glass plates) and observation of plates was carried out under a UV CAMAG spectrometer (254 nm).

Plant Material. The plant material (*F. diversivittata* Regel & Schmalh.-Rech.) was collected from the Hezarmasjed mountains, Khorasan Razavi province, Iran, in May 2006. The plant material was identified by Mohammad Reza Joharchi, Ferdowsi University of Mashhad Herbarium (FUMH).

A voucher specimen (No. 1006) has been deposited at the Herbarium of the Faculty of Pharmacy, Mashhad University of Medical Sciences.

Extraction and Isolation. The air-dried roots (350 g) were ground into a powder and extracted exhaustively by maceration at room temperature with CH_2Cl_2. After filtration, the extract was concentrated under vacuum to yield 15 g of a brown residue.

The extract (15 g) was subjected to column chromatography on silica gel (5 × 60 cm) using petrol with increasing volumes of EtOAc (petrol–EtOAc (20:1, 2.1 L), (15:1, 3.2 L) and (10:1, 3.3 L)). The fractions (200 mL each) were compared by TLC (silica gel using petrol–EtOAc, different ratios), and those giving similar spots were combined. The first fraction that eluted from the column contained diversivittatin as a yellow oil (122.9 mg).

Diversivittatin (1): yellow oil; UV (λ_{max}, CH_2Cl_2, nm): 210; IR (ν_{max}, CH_2Cl_2, cm^{-1}): 2927, 1716 (ester), 1636, 1456, 1433, 1231, 1147, 1091, 1043; HR-EI-MS *m/z* 292.1280, calcd 292.1311.

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