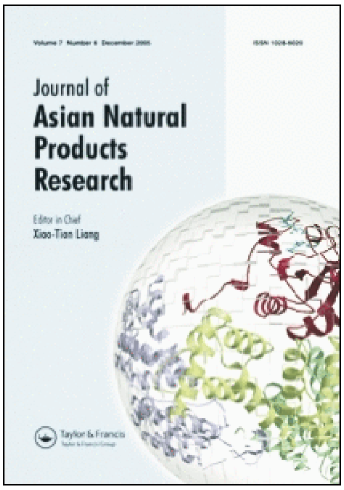

Journal of Asian Natural Products Research
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713454007

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To cite this Article Auzi, Abdurazag A. , Gray, Alexander I. , Salem, Mohamed M. , Badwan, Adnan A. and Sarker, Satyajit D.(2008) 'Feruhermonins A-C: three daucane esters from the seeds of *Ferula hermonis* (Apiaceae)', Journal of Asian Natural Products Research, 10: 8, 701 — 707

To link to this Article: DOI: 10.1080/10286020802016040

URL: http://dx.doi.org/10.1080/10286020802016040

Journal of Asian Natural Products Research
Vol. 10, No. 8, August 2008, 701–707

Feruhermonins A–C: three daucane esters from the seeds of *Ferula hermonis* (Apiaceae)

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(*Received 13 March 2007; final version received 27 July 2007*)

Seventeen daucane esters have been isolated from the seeds of *Ferula hermonis* Boiss (Apiaceae). Three of these sesquiterpenes, 4β-hydroxy-6α-benzoyl-7-daucen-9-one (**1**), 4β, 8β-dihydroxy-6α-benzoyl-dauc-9-ene (**2**), and 4β, 9α-dihydroxy-6α-benzoyl-dauc-7-ene (**4**), named feruhermonins A–C, respectively, are novel natural products. The structures of these compounds were elucidated unequivocally by a series of 1D and 2D NMR analyses. Although 4β, 8β-dihydroxy-6α-(4-hydroxy-3-methoxybenzoyl)-dauc-9-ene (**3**) was reported previously, the complete spectroscopic data for this compound are presented here for the first time.

Keywords: *Ferula hermonis*; seeds; Apiaceae; daucane esters; sesquiterpene

1. Introduction

Ferula hermonis Boiss (Apiaceae), commonly known as 'Shilsh-el-zallouh' or 'Hashishat-al-kattira', is a small shrub that grows abundantly on the Hermon Mountain between Syria and Lebanon [1,2]. This plant has long been used in the Middle East as an aphrodisiac, and for the treatment of frigidity and impotence [1,3]. Previous phytochemical studies, carried out on the root of this plant, have revealed the presence of various sesquiterpenes, mainly of daucane type [4,5]. We now report on the isolation and structure elucidation of 17 daucane esters from the seeds of *F. hermonis,* 3 of which (**1**, **2**, and **4**) are new sesquiterpene esters.

2. Results and discussion

A combination of vacuum liquid chromatography (VLC), gel filtration, and preparative thin layer chromatography (PTLC) of the *n*-hexane–EtOAc (1:1) extract of the seeds of *F. hermonis* afforded 17 daucane sesquiterpenes (**1**–**17**), 3 of which (**1**, **2**, and **4**) are new sesquiterpene esters. The structures of all compounds were elucidated by UV, IR, MS, and extensive 1D and 2D NMR analyses.

Thirteen known daucane sesquiterpenes (**5**–**17**) were identified as 4β, 9α-dihydroxy-6α-(4-hydroxy-3-methoxybenzoyl)-dauc-7-ene (**5**) [6], lancerodiol 4-hydroxybenzoate (**6**) [7], 9-*epi*-6α-(4-hydroxybenzoyl)-isolancerotriol (ferugin, **7**) [8], isolancerotriol 6α-vanillate (**8**) [9], jaeschkeanadiol (**9**) [10], teferidine (**10**) [10,11], ferutinin (**11**) [10], 14-(4-hydroxybenzoyloxy)-dauca-4,8-diene (**12**) [4], 14-(4-hydroxy-3-methoxy-benzoyloxy)-dauca-4,8-diene (**13**) [4], 2, 3-epoxyjaeschkeanadiol (**14**) [12], 2,3-epoxyjaeschkeanadiol 4-hydroxybenzoate (**15**) [12],

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ISSN 1028-6020 print/ISSN 1477-2213 online

DOI: 10.1080/10286020802016040
http://www.informaworld.com

2,3-epoxy-jaeschkeanadiol vanillate (**16**) [12], and vaginatin (**17**) [13]. All the spectral data of these compounds were in good agreement with the respective published data.

Analysis of the ^{1}H- and ^{13}C-NMR spectra of compounds **1**–**4** (Tables 1 and 2; Figure 1) revealed the presence of daucane (or substituted carotane) nucleus, with an isopropyl moiety at C-4 and a benzoyl (in **1**, **2**, and **4**) or substituted benzoyl (in **3**) ester at C-6 [14]. All the carotane sesquiterpenes isolated from the genus *Ferula* are hydroxylated at C-6 and have an α-stereochemistry at C-5 [7]. The MS spectra of **1**–**4** confirmed the presence of isopropyl group, evident from the loss of the C_3H_7 fragment, which is typical for carotane sesquiterpenes with a hydroxyl function at C-4 [15]. The IR spectra of these compounds revealed strong absorption bands corresponding to the hydroxyl and ester groups.

The HR-FAB-MS of **1** gave a *pseudo*molecular ion peak at *m/z* 357.2066 $[M + H]^+$, corresponding to the empirical formula $C_{22}H_{29}O_4$. In the ^{13}C-NMR spectrum of **1** (Table 2), in addition to the signals associated with the common nucleus as outlined above, a signal at δ_C 201.3 indicated the presence of a ketonic carbonyl (oxo) functionality. The placement of the carbonyl group at C-9 was established by means of long-range ^{1}H–^{13}C correlations observed in its HMBC spectrum (Table 3). The C-14 methyl signal (δ_H 1.90) displayed ^{3}J correlations with the carbonyl carbon (C-9, δ_C 201.3) and the olefinic methine (C-7, δ_C 138.4), and a ^{2}J correlation with the olefinic quaternary carbon (C-8, δ_C 136.2). Moreover, the methylene protons at C-10 (δ_H 2.75 and 2.62) correlated (^{3}J) with the methyl carbon at C-15 (δ_C 20.5) and ^{2}J to the C-9 carbonyl. The other correlations observed in the HMBC spectrum were consistent with the dauc-7-ene nucleus as depicted in **1**, which is a novel sesquiterpene ester, 4β-hydroxyl-6α-benzoyl-7-daucen-9-one (named feruhermonin A). The relative stereochemistry at the chiral centers was determined from the NOE interactions observed in the phase-sensitive NOESY spectrum (Table 4). All spectroscopic data of **1** were comparable with that of a similar compound, 4β-hydroxy-6α-(4-hydroxy-3-methoxybenzoyl)-7-daucen-9-one, isolated

Table 1. ^{1}H-NMR spectral data of sesquiterpenes **1**–**4**.

	Chemical shits (δ) in ppm			
Position	**1**	**2**	**3**	**4**
2	1.40 m; 1.60 m	1.41 m; 1.64 m	1.40 m; 1.70 m	1.33 m; 1.63 m
3	1.95 m; 2.05 m	1.60 m; 1.98 m	1.50 m; 1.70 m	1.63 n; 1.69 m
5	2.52 d (10.8)	2.51 d (10.6)	2.42 d (10.7)	2.23 d (10.8)
6	6.13 m	5.87 ddd (10.6, 5.4, 1.8)	5.87 ddd (10.7, 5.1, 1.9)	5.92 m
7	6.19 d (2.5)	2.54 dd (15.3, 5.4) 2.14 dt (15.3, 1.8)	2.81 dd (15.3, 5.1) 2.09 dt (15.3, 1.9)	5.39 br s
9	–	5.50 dd (11.6, 1.8)	5.50 dd (11.8, 1.9)	4.31 m
10	2.62 m; 2.75 m	5.59 d (11.6)	5.79 d (11.8)	1.65 m; 2.12 m
11	1.82 q (6.8)	1.60 sep (6.8)	1.70 m	1.82 m
12	0.85 d (6.8)	0.86 d (6.8)	0.89 d (6.8)	0.93 d (6.8)
13	0.97 d (6.8)	0.87 d (6.8)	0.89 d (6.8)	0.85 d (6.8)
14	1.90 s	1.63 s	1.56 s	1.90 s
15	1.26 s	1.30 s	1.32 s	1.21 s
2′	8.07 m	8.04 m	7.55 d (1.8)	8.06 m
3′	7.49 m	7.48 m	–	7.47 m
4′	7.62 m	7.61 m	–	7.60 m
5′	7.49 m	7.48 m	6.96 d (8.3)	7.47 m
6′	8.07 m	8.04 m	7.58 dd (8.3, 1.8)	8.06
3′-OMe	–	–	3.94 s	–

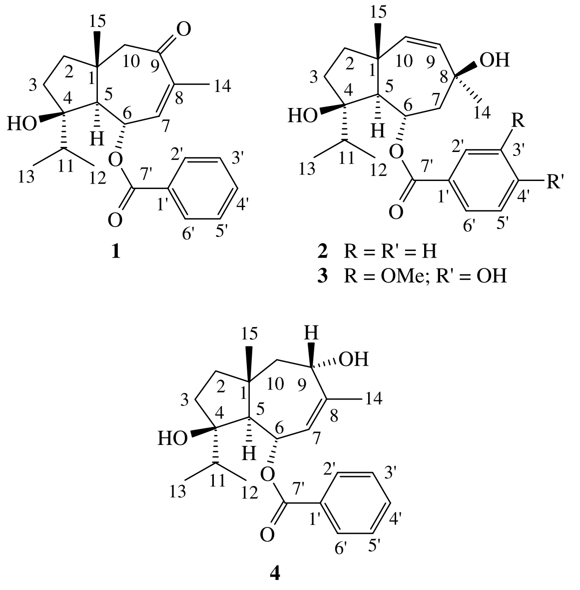
Spectra obtained in $CDCl_3$.

Table 2. ^{13}C-NMR spectral data of sesquiterpenes **1**–**4**.

	Chemical shits (δ) in ppm			
Position	**1**	**2**	**3**	**4**
1	56.6	46.0	46.1	42.7
2	42.4	41.5	41.5	42.2
3	32.5	31.2	31.2	32.1
4	86.8	85.8	85.7	86.7
5	54.8	52.3	52.8	55.0
6	73.0	71.1	70.8	73.5
7	138.4	46.3	44.6	126.3
8	136.2	73.0	71.7	139.4
9	201.3	134.9	132.4	70.0
10	41.5	138.3	141.8	50.7
11	37.1	36.8	36.9	37.2
12	17.5	18.6	18.7	18.6
13	18.5	17.6	17.6	17.6
14	22.0	29.2	29.2	24.1
15	20.5	21.5	21.6	19.2
1′	130.1	130.5	122.6	130.5
2′	130.0	129.9	112.1	130.0
3′	128.8	128.5	146.3	128.7
4′	133.7	133.5	150.6	133.4
5′	128.8	133.5	114.5	128.7
6′	130.0	128.5	124.5	130.0
7′	166.5	167.1	166.9	166.9
3′-OMe	–	–	56.3	–

from *F. orientalis* [16], with the only exception that the data of **1** corresponded to the presence of a benzoyl moiety, instead of a 4-hydroxy-3-methoxybenzoyl moiety.

The HR-EI-MS spectrum of **2** showed the molecular ion peak at *m/z* 358.2144 $[M]^+$, corresponding to the molecular formula $C_{22}H_{30}O_4$. The dauc-9-ene nucleus was deduced from the HMBC experiment (Table 3), where $^1H-^{13}C$ long-range correlations were observed between H-9 (δ 5.50) and C-1 (δ 46.0), C-10 (δ 138.3), C-14 (δ 29.2) and C-7 (δ 46.3), and H-10 (δ 5.59) and C-1, C-2 (δ 41.5), C-8 (δ 73.0) and C-5 (δ 52.3). In the ^{13}C-NMR spectrum (Table 2), in addition to the signals associated with the dauc-9-ene skeleton, there was a signal for an oxygenated quaternary carbon (δ 73.0), which correlated with the C-14 methyl (δ 1.63), H-6 (δ 5.87), H-9, and H-10 proton signals in the HMBC spectrum. Thus, the additional hydroxyl group could be placed at C-8. The other correlations observed in the HMBC spectrum unambiguously supported the structure **2**, which is a new sesquiterpene ester, 4β, 8β-dihydroxy-6α-benzoyl-dauc-9-ene (named feruhermonin B). The relative stereochemistry



2 R = R' = H
3 R = OMe; R' = OH

4

Figure 1. Structures of compounds **1**–**4**.

Table 3. Key ^{1}H–^{13}C long-range correlations observed in the HMBC spectra of **1**–**4**.

	^{1}H–^{13}C long-range HMBC correlations							
	1		**2**		**3**		**4**	
Position	^2J	^3J	^2J	^3J	^2J	^3J	^2J	^3J
2	C-1, C-3	C-4, C-5, C-10, C-15	C-1, C-3	C-4, C-5, C-10, C-15	C-1, C-3	C-4, C-5, C-10, C-15	C-1, C-3	C-4, C-5, C-10, C-15
3	C-2, C-4	C-1, C-5, C-11	C-2, C-4	C-1, C-5, C-11	C-2, C-4	C-1, C-5, C-11	C-2, C-4	C-1, C-5, C-11
5	C-1, C-4, C-6	C-2, C-3, C-7, C-10 C-11, C-15	C-1, C-4, C-6	C-2, C-3, C-7, C-10 C-11, C-15	C-1, C-4, C-6	C-2, C-3, C-7, C-10 C-11, C-15	C-1, C-4, C-6	C-2, C-3, C-7, C-10, C-11 C-15
6	C-5, C-7	C-1, C-4, C-8, C-7′	C-5, C-7	C-1, C-4, C-8, C-7′	C-5, C-7	C-1, C-4, C-8, C-7′	C-5, C-7	C-1, C-4, C-8, C-7′
7	C-6, C-8	C-5, C-9, C-14	C-6, C-8	C-5, C-9, C-14	C-6, C-8	C-5, C-9, C-14	C-6, C-8	C-5, C-9, C-14
9	–	–	C-8, C-10	C-1, C-7, C-14	C-8, C-10	C-1, C-7, C-14	C-8, C-10	C-1, C-7, C-14
10	C-1, C-9	C-2, C-5, C-8, C-15	C-1, C-9	C-2, C-5, C-8, C-15	C-1, C-9	C-2, C-5, C-8, C-15	C-1, C-9	C-2, C-5, C-8, C-15
11	C-4, C-12, C-13	C-3, C-5	C-4, C-12, C-13	C-3, C-5	C-4, C-12, C-13	C-3, C-5	C-4, C-12, C-13	C-3, C-5
12	C-11	C-4, C-13	C-11	C-4, C-13	C-11	C-4, C-13	C-11	C-4, C-13
13	C-11	C-4, C-12	C-11	C-4, C-12	C-11	C-4, C-12	C-11	C-4, C-12
14	C-8	C-7, C-9	C-8	C-7, C-9	C-8	C-7, C-9	C-8	C-7, C-9
15	C-1	C-2, C-5, C-10	C-1	C-2, C-5, C-10	C-1	C-2, C-5, C-10	C-1	C-2, C-5, C-10
2′	C-1′, C-3′	C-4′, C-6′, C-7′	C-1′, C-3′	C-4′, C-6′, C-7′	C-1′, C-3′	C-4′, C-6′, C-7′	C-1′, C-3′	C-4′, C-6′, C-7′
3′	C-2′, C-4′	C-1′, C-5′	C-2′, C-4′	C-1′, C-5′	–	–	C-2′, C-4′	C-1′, C-5′
4′	C-3′, C-5′	C-2′, C-6′	C-3′, C-5′	C-2′, C-6′	–	–	C-3′, C-5′	C-2′, C-6′
5′	C-4′, C-6′	C-1′, C-3′	C-4′, C-6′	C-1′, C-3′	C-4′, C-6′	C-1′, C-3′	C-4′, C-6′	C-1′, C-3′
6′	C-1′, C-5′	C-2′, C-4′, C-7′	C-1′, C-5′	C-2′, C-4′, C-7′	C-1′, C-5′	C-2′, C-4′, C-7′	C-1′, C-5′	C-2′, C-4′, C-7′
3′-OMe	–	–	–	–	–	C-3′	–	–

Table 4. Key NOE interactions observed in the $^1H-^1H$ NOESY spectra of **1**–**4**.

	$^1H-^1H$ NOE interactions			
Proton signal	**1**	**2**	**3**	**4**
H-5	H-11, Me-12, Me-13	H-7α, H-11, Me-12, Me-13, Me-14	H-7α, H-11, Me-12, Me-13	H-11, Me-12, Me-13
H-6	Me-15	Me-15, H-7β	Me-15, H-7β	H-9, Me-15
H-7	Me-14	–	–	Me-14
H-7α	–	H-5, Me-14	H-5, Me-14	–
H-7β	–	H-6, Me-15	H-6, Me-15	–
H-9	–	H-10	H-10	H-6, Me-15
H-10	–	H-9	H-9	–
H-11	H-5	H-5	H-5	H-5
Me-12	H-5, Me-13	H-5, Me-13	H-5, Me-13	H-5, Me-13
Me-13	H-5, Me-12	H-5, Me-12	H-5, Me-12	H-5, Me-12
Me-14	H-7	H-5, H-7a	H-5, H-7a	H-7
Me-15	H-6	H-6	H-6	H-6
H-2′	–	–	3′-OMe	–
3′-OMe	–	–	H-2′	–

at the chiral centers was determined from the NOE interactions observed in the phase-sensitive NOESY spectrum (Table 4).

The ^{1}H- and ^{13}C-NMR spectra (Tables 1 and 2) of **3** were similar to those of **2** with the only exception that instead of the signals associated with a benzoyl moiety, there were signals corresponding to a 4-hydroxy-3-methoxybenzoyl moiety. The HR-EI-MS analysis of **3** confirmed the molecular formula $C_{23}H_{32}O_6$ by displaying the molecular ion peak at *m/z* 404.2199. Thus, compound **3** was identified as 4β, 8β-dihydroxy-6α-(4-hydroxy-3-methoxybenzoyl)-dauc-9-ene. Although Chen *et al.*[6] mentioned this compound in their article, there were no data or appropriate reference cited. Moreover, search of the relevant databases, e.g. *Combined Chemical Dictionary* [17], did not produce any match for this compound.

The HR-EI-MS of **4** showed the molecular ion peak at *m/z* 358.2144, corresponding to the empirical formula $C_{22}H_{30}O_4$. The ^{1}H- and ^{13}C-NMR spectra of **4** were similar to those of **1** with the exception that an oxymethine signal (δ_H 4.31, δ_C 70.0) (in **4**) replaced the ketonic carbonyl signal (in **1**). In the HMBC spectrum of **4** (Table 3), the long-range $^1H-^{13}C$ correlations between the methyl protons at C-14 (δ 1.90) and the olefinic carbons at C-7 (δ 126.3) and C-8 (δ 139.4), and the oxymethine carbon atom at C-9 (δ 70.0), confirmed the placement of the additional hydroxyl group at C-9. The other correlations observed in the HMBC spectrum unambiguously supported the structure **4**, which is a new sesquiterpene ester, 4β, 9α-dihydroxy-6α-benzoyl-dauc-7-ene (named feruhermonin C). The relative stereochemistry at the chiral centers was determined from the NOE interactions observed in the phase-sensitive NOESY spectrum (Table 4). All spectroscopic data of **4** were comparable with those of a similar compound, 4β, 9α-dihydroxy-6α-(4-hydroxy-3-methoxybenzoyl)-dauc-7-ene, isolated from *F. kuhistanica* [6] with the only exception that the data of **4** corresponded to the presence of a benzoyl moiety, instead of a 4-hydroxy-3-methoxybenzoyl moiety.

All compounds (**1**–**17**) isolated from the seeds of *F. hermonis* conform to the general structural features of the daucane sesquiterpenoids found in the species of the genus *Ferula* [18].

3. Experimental

3.1 General experimental procedures

UV spectra were obtained using a Hewlett-Packard 8453 UV/vi spectrophotometer in

MeOH. IR spectra (KBr) were taken on a JASCO FTIR-4000/6000 Spectrometer. NMR spectra were recorded in $CDCl_3$ on a Bruker AMX 400 MHz NMR Spectrometer (400 MHz for ^1H and 100 MHz for ^{13}C) using the residual solvent peaks as internal standard. MS analyses were performed on a Finnigan MAT 95 spectrometer. Merck Silica gel 60 H was used for VLC. HMBC spectra were optimized for a long-range J_{H-C} of 9 Hz, and NOESY experiment was carried out with a mixing time of 0.8 s.

3.2 Plant material

The seeds of *Ferula hermonis* were collected in October 2000 from the Mount Hermon at the joint borders of Lebanon and Syria. The plant material was identified by the Botany Department, University of El-Fateh, and a voucher specimen (FHS1999) has been maintained in their herbarium.

3.3 Extraction and isolation

The ground seeds (500 g) of *F. hermonis* were macerated with 1 l of *n*-hexane–EtOAc (1:1) for 24 h. The filtered extract was concentrated under reduced pressure to obtain oily substance (105 g). A portion (40 g) of the oil was subjected to silica gel VLC using *n*-hexane and EtOAc with increasing polarity. The 10% EtOAc in *n*-hexane fraction was passed through gel filtration using Sephadex LH-20, eluting with chloroform. Preparative TLC, using *n*-hexane–EtOAc of various proportions as a mobile phase, of the fractions obtained from the gel filtration resulted in the isolation of **1–17.**

3.3.1 4β-Hydroxy-6α-benzoyl-7-daucen-9-one (feruhermonin A) (*1*)

Oil; 12.1 mg; UV λ_{max} (MeOH): 232, 263, and 294 nm; IR ν_{max} (KBr) cm^{-1}: 3500, 2917, 1715, 1666, 1451, 1373, 1271, 1177, 1108, 1069, 1026, and 712; ^1H-NMR (400 MHz, $CDCl_3$) (Table 1) and ^{13}C-NMR (100 MHz, $CDCl_3$) (Table 2); and HR-FAB-MS *m/z* 357.2065 $[M + H]^+$ (calcd for $C_{22}H_{29}O_4$, 357.2066).

3.3.2 4β, 8β-Dihydroxy-6α-benzoyl-dauc-9-ene (feruhermonin B) (*2*)

Oil; 9.1 mg; UV λ_{max} (MeOH): 220, 262, and 290 nm; IR ν_{max} (KBr) cm^{-1}: 3550, 3200, 1750, 1680, 1520, 1450,1375, 1250, 1130, 1108, 1069, 1020, and 710; ^1H-NMR (400 MHz, $CDCl_3$) (Table 1) and ^{13}C-NMR (100 MHz, $CDCl_3$) (Table 2); HR-EI-MS *m/z* 358.2144 $[M]^+$ (calcd for $C_{22}H_{30}O_4$, 358.2144); and EI-MS *m/z* (rel. int.): 358 (20), 357 (15), 315 (28), and 298 (17).

3.3.3 4β, 8β-Dihydroxy-6α-(4-hydroxy-3-methoxybenzoyl)-dauc-9-ene (*3*)

Oil; 8.7 mg; UV λ_{max} (MeOH): 208, 221, 260, and 292 nm; IR ν_{max} (KBr) cm^{-1}: 3600, 3220, 1750, 1680, 1520, 1450, 1370, 1250, 1130, 1108, 1120, and 720; ^1H-NMR (400 MHz, $CDCl_3$) (Table 1) and ^{13}C-NMR (100 MHz, $CDCl_3$) (Table 2); HR-EI-MS *m/z* 404.2198 $[M]^+$ (calcd for $C_{23}H_{32}O_6$, 404.2199); and EI-MS *m/z* (rel. int.): 404 (20), 403 (43), 386 (15), 361 (28), and 290 (17).

3.3.4 4β, 9α-Dihydroxy-6α-benzoyl-dauc-7-ene (feruhermonin C) (*4*)

Oil; 10.5 mg; UV λ_{max} (MeOH): 210, 222, 262, and 290 nm; IR ν_{max} (KBr) cm^{-1}: 3500, 3100, 1750, 1660, 1552, 1450, 1372, 1260, 1130, 1108, 1070, 1020, and 710; ^1H-NMR (400 MHz, $CDCl_3$) (Table 1) and ^{13}C-NMR (100 MHz, $CDCl_3$) (Table 2); HR-EI-MS *m/z* 358.2142 $[M]^+$ (calcd for $C_{22}H_{30}O_4$, 358.2144); and EI-MS *m/z* (rel. int.): 358 (20), 357 (15), 341 (15), 315 (37), and 300 (25).

Acknowledgements

We thank Mrs A.B. Bhasin (Strathclyde University) for MS analyses. NMR studies were performed in the Strathclyde University NMR Laboratories, Glasgow, UK.

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