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Polar secondary metabolites of *Ferula persica* roots

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Abstract

Phytochemical investigation of the methanolic extract of the dried roots of *Ferula persica* resulted in four sesquiterpene coumarin glycosides, persicaosides A–D, and two known phytosterol glucosides, sitosterol 3-*O*-β-glucoside and stigmasterol 3-*O*-β-glucoside. The structures of these compounds were elucidated by extensive spectroscopic methods including 1D-(^{1}H and ^{13}C) and 2D NMR experiments (DQF-COSY, HSQC, HMBC, and ROESY) as well as ESIMS and TOFMS analyses.
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Keywords: *Ferula persica*; Apiaceae; Persicaosides; Roots; Sesquiterpene coumarin glycosides

1. Introduction

The genus *Ferula* (Apiaceae) is represented by more than 150 species, and members of this genus are widespread throughout central Asia (Pimenov and Leonov, 2004). Several species of the genus *Ferula* are used in traditional medicine. The chemical constituents of plants in the genus *Ferula* have been studied by many groups (Murray et al., 1982). The compounds typically found in this genus are sesquiterpenes (Valle et al., 1987; Miski and Jakupovic, 1990), sesquiterpene coumarins (Abd El-Razek et al., 2003), and sulphur-containing compounds (Al-said et al., 1996; Iranshahi et al., 2003a). A few sesquiterpene coumarin glycosides have also been reported from *Ferula* species (Abd El-Razek et al., 2003). Up to now, almost all research has focused on identifying non-polar constituents of *Ferula* species (Abd El-Razek et al., 2003). The roots of *Ferula persica* have been used in folk medicine to treat diabetes (Afifi and Abu-Irmaileh, 2000). The chemistry of *F. persica* has previously been studied and different non-polar components from the aerial parts (Iranshahi et al., 2003b) and the roots (Iranshahi et al., 2003a, 2004) of the plant have been identified. However, the polar constituents of this medicinal plant have not been determined. Here we report the isolation and structure elucidation of four new sesquiterpene glycosides from a methanol extract of the roots of *F. persica*.

2. Results and discussion

Dried, powdered roots of *F. persica* (250 g) were sequentially extracted with chloroform and methanol. The methanol extract was fractioned initially by silica gel column chromatography, and further separation was carried out by reverse-phase (C_{18}) preparative thin layer chromatography to give sesquiterpene coumarin glycosides (Fig. 1).

Compound **1** was determined to be a sesquiterpene coumarin glycoside by the presence of diagnostic peaks in the ^{1}H NMR and ^{13}C NMR spectra (Tables 1 and 2). The ^{13}C NMR spectrum of compound **1** displayed 36 carbon signals, nine being typical of an umbelliferone skeleton,

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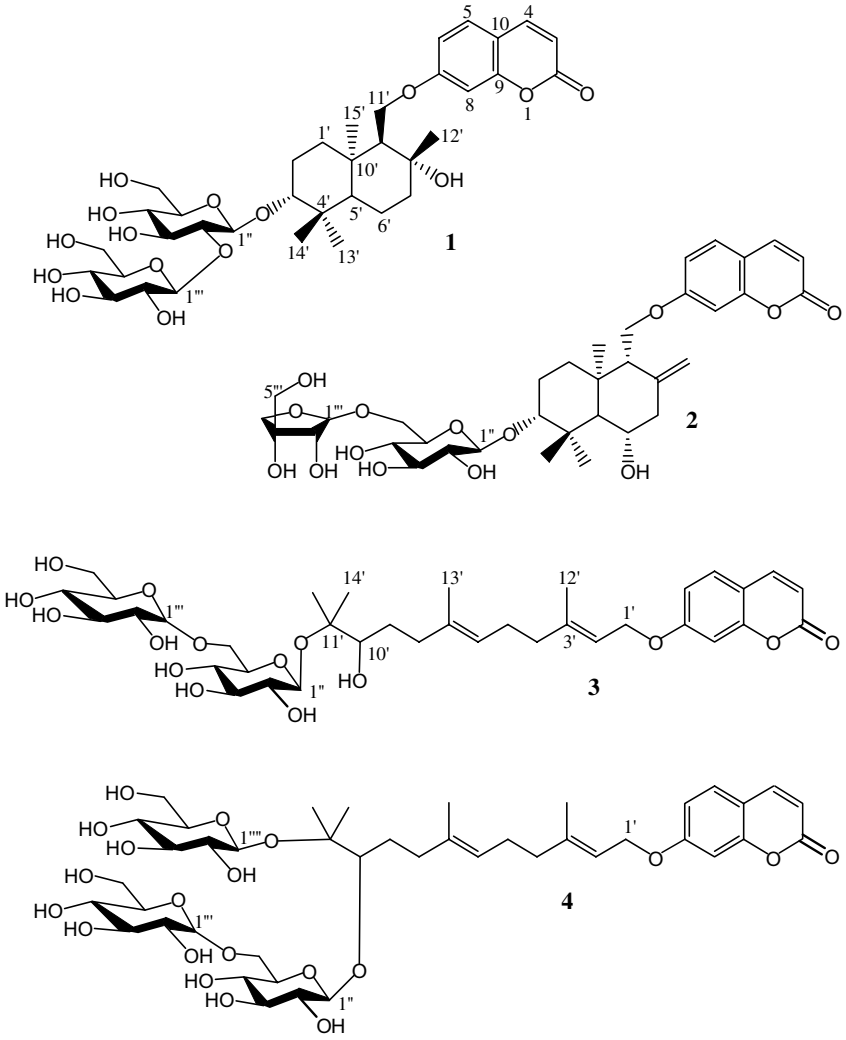

Fig. 1. Compounds **1**–**4** isolated from *Ferula persica*.

15 signals ascribable to a sesquiterpene moiety, and 12 signals to a sugar moiety. HSQC spectrum classified the carbon signals to four aliphatic methylenes at δ_C 19.5, 24.0, 36.7 and 39.9, to a primary alcoholic carbon at δ_C 69.1 characteristic for C-11′, to ten methines, five of them for umbelliferone moiety at δ_C 113.4 (C-3), 145.8 (C-4), 130.5 (C-5), 114.3 (C-6) and 102.1 (C-8) and to four methyls at δ_C 17.2, 24.9, 29.2, and 31.4. The ^{1}H NMR and ^{13}C NMR (CD_3OD, Tables 1 and 2) spectra of **1**, which were assigned by HSQC, HMBC, and COSY experiments, show signals assignable to two β-glucopyranosyl moieties [δ 4.43 (d, $J = 7.5$ Hz, H-1″) and 4.49 (d, $J = 7.5$ Hz, H-1‴)], together with an aglycone moiety. Long-range HMBC correlations from the proton signal at δ_H 1.26 (Me-12′) to the carbon resonance at δ_C 58.8 (C-9′) and from the protons of the primary oxygenated carbon C-11′ to the same carbon C-9′ revealed the location of a tertiary methyl group (Me-12′) at C-8′. A third HMBC correlation between the singlet methyl (Me-15′) at δ_H 1.37 and the carbon resonance at δ_C 58.8 (C-9′) allowed a singlet methyl to be assigned at C-10′. The remaining methyl groups (Me-13′ and Me-14′) were established at C-4′ from the HMBC correlations between the carbon resonance at δ_C 39.5 (C-4′), and the proton signals at δ_H 0.86 (Me-14′) and 1.08 (Me-13′). The inter-glycosidic linkage was defined on the basis of the long-range correlations between C-1‴ and H-2″ and between C-2″ and H-1‴. The ROESY experiment supported the relative configuration of the stereogenic centers at C-3′, C-5′, C-8′, C-9′, and C-10′. In particular ROEs H-11′/H-5′ and Me-13′/Me-15′, H-3′/H-5′, Me-12′/CH_2-11′ established an α-orientation for both Me-13′ and Me-15′, and a β-orientation for H-3′, H-5′, H-11′, Me-12′ and Me-14′. The main ROEs effects observed are depicted in Fig. 2. ESIMS analysis of **1** showed the positive and negative ion peaks at *m/z* 747 $[M+Na]^+$ and 759 $[M+Cl]^-$, corresponding to the molecular formula of $C_{36}H_{52}O_{15}$ for **1**, respectively. On the basis of the above results, the structure of **1** was elucidated as 3′-*O*-[β-glucopyranosyl-(1 → 2)-β-glucopyranosyl]ferukrin and named persicaoside A.

Compound **2** was determined to be a sesquiterpene coumarin glycoside by the presence of diagnostic peaks in the ^{1}H NMR and ^{13}C NMR spectra (Tables 1 and 2). The

Table 1
^{1}H NMR data (δ_H) for compounds **1–4** (500 MHz, MeOH-d_4, mult., J in Hz)

Position	**1**	**2**	**3**	**4**
2	–	–	–	–
3	6.23 *d* (9.5)	6.24 *d* (9.5)	6.25 *d* (9.5)	6.25 *d* (9.5)
4	7.87 *d* (9.5)	7.86 *d* (9.5)	7.88 *d* (9.5)	7.88 *d* (9.5)
5	7.52 *d* (9.5)	7.50 *d* (9)	7.52 *d* (8.5)	7.55 *d* (8.5)
6	6.90 *dd* (9.5, 1.5)	6.89 *d* (9)	6.91 *dd* (8.5, 2.5)	6.86 *dd* (8.5, 2.5)
8	6.91 *d* (1.5)	6.90 *s*	6.88 *d* (2.5)	6.84 *d* (2.5)
1′	*eq* 1.40 *dt* (13.5, 3.5)	*ax* 1.48 *m*	4.66 *d* (6.5)	4.68 *d* (6.5)
	ax 1.47 *dt*[a]	*eq* 1.79 *m*		
2′	1.80 *m*	1.66 *m*	5.46 *t* (6.5)	5.48 *t* (6.5)
3′	3.32[a]	3.17[a]		
4′			2.12 *t* (6.5)	2.12 *t* (6.5)
5′	1.47[a]	1.45 *d* (10.5)	2.17 *t* (6.5)	2.17 *t* (6.5)
6′	*eq* 1.56 *m*	4.07 *ddd* (10.5, 5.0, 5.0)	5.15 *t* (6)	5.15 *t* (6)
	ax 1.75 *m*			
7′	*eq* 1.67 *dd* (13.5, 4)	*ax* 2.17 *dd* (13, 5) like *t*		
	ax 1.74[a]	*eq* 2.91 *dd* (13, 5)		
8′			1.97 *m*	1.97 *m*
			2.21 *m*	2.21 *m*
9′	1.56 *dd* (3.5, 2.5) like brs	2.29 *dd* (7.5, 4.5)	1.35 *m*	1.35 *m*
			1.57 *m*	1.57 *m*
10′			3.43 *dd* (10.5, 1.5)	3.48 *dd* (10.5, 1.5)
11′	4.15 *dd* (11, 3.5)	4.21 *dd* (9.5, 7.5)		
	4.17 *dd* (11, 2.5)	4.27 *dd* (9.5, 4)		
12′	1.26 *s*	4.62, 4.97 brs	1.78 *s*	1.78 *s*
13′	0.86 *s*	1.30 *s*	1.61 *s*	1.60 *s*
14′	1.08 *s*	1.06 *s*	1.18 *s*	1.19 *s*
15′	1.37 *s*	0.91 *s*	1.21 *s*	1.26 *s*
1″	4.43 *d* (7.5)	4.39 *d* (8)	4.49 *d* (8)	4.59 *d* (8)
2″	3.40 *dd* (9, 7.5)	3.40 *m*	3.15 *t* (8)	3.25 *dd* (9.5, 8)
3″	3.50[a]	3.17 *m*	3.46[a]	3.37[a]
4″	3.30[a]	3.30[a]	3.31[a]	3.28[a]
5″	3.16[a]	3.37[a]	3.35[a]	3.31[a]
6″	3.60 *dd* (12, 6)	3.57 *dd* (11, 2)	3.71 *dd* (11.5, 6)	3.74 *dd* (11.5, 5.5)
	3.79 *dd* (12, 5)	3.98 *dd* (11, 2)	4.11 *dd* (11.5, 2)	4.11 *dd* (11.5, 2)
1‴	4.49 *d* (7.5)	4.97 *d* (1.5)	4.33 *d* (8)	4.35 *d* (8)
2‴	3.23[a]	3.87 *d* (1.5)	3.20 *dd* (9, 8)	3.20 *dd* (9.5, 8)
3‴	3.36[a]		3.35[a]	3.37[a]
4‴	3.36[a]	3.74 *d* (10)	3.27[a]	3.30[a]
		3.93 *d* (10)		
5‴	3.24[a]	3.58 *s*	3.27[a]	3.28[a]
6‴	3.71 *dd* (12, 5)		3.66 *dd* (11.5, 5)	3.66 *dd*[a]
	3.81 *dd* (12, 2.5)		3.85 *dd* (11.5, 2.5)	3.85 *dd*[a]
1⁗				4.61 *d* (7)
2⁗				3.41 *dd* (9, 7)
3⁗				3.37[a]
4⁗				3.36[a]
5⁗				3.58[a]
6⁗				3.64 dd[a]
				3.84 dd[a]

[a] Overlapped with other signals.

NMR spectroscopic data of **2** were similar to those of **1**, the main differences between the two compounds being the signals assigned to the sesquiterpene and the sugar units. Regarding this portion and with respect to persicaoside A, the HSQC spectrum shows the occurrence of three tertiary methyls and one olefinic methylene, while the HMBC spectrum revealed the occurrence of two oxygenated methines and one olefinic quaternary carbon at C-3′, C-6′ and C-8′, respectively. The ^{1}H NMR and ^{13}C NMR spectra (CD_3OD, Tables 1 and 2) of **2** show signals assignable to a sesquiterpene coumarin moiety [δ 1.30, 1.06, 0.91 (all *s*, H_3-13, 14, and 15), 1.56 (*dd*-like brs, H-9′), 3.17 (H-3′), 4.07 (ddd, H-6′), 4.21, 4.29 (both *dd*, H_2-11′), 4.62, 4.97 (both *s*, H_2-12′) 6.24, 7.86, 7.50, 6.89 (all *d*, H-3, 4, 5, and 6, see Table 1 for coupling constants)], one glucopyranosyl moiety [δ 4.43 (*d*, $J = 7.5$ Hz, H-1″)], and one apiofuranosyl moiety [δ 4.97 (*d*, $J = 1.5$, H-1‴), 3.87 (*d*, $J = 1.5$, H-2‴, 3.74 and 3.93 (both *d*, $J = 10$, H_2-4‴, and

Table 2
^{13}C NMR data (δ_C) for compounds **1–4** (125 MHz, MeOH-d_4).

Position	1	2	3	4
2	163.4	163.3	163.3	163.5
3	113.4	113.3	113.2	113.2
4	145.8	145.8	145.9	145.9
5	130.5	130.4	130.4	130.4
6	114.3	114.3	114.5	114.5
7	163.5	163.9	163.9	163.8
8	102.1	101.3	102.5	102.5
9	157.2	157.2	157.1	157.1
10	114.1	114.2	113.9	113.9
1′	36.7	38.6	66.6	66.6
2′	24.0	28.1	120.4	120.4
3′	85.8	79.6	142.9	142.9
4′	39.5	40.9	40.5	40.5
5′	50.0	59.2	27.1	27.1
6′	19.5	76.6	125.1	125.1
7′	39.9	44.0	136.5	136.5
8′	73.9	145.3	37.8	37.8
9′	58.8	55.8	31.0	31.0
10′	38.9	39.4	77.4	83.9
11′	69.1	67.1	81.9	82.4
12′	31.4	109.6	16.8	16.8
13′	29.2	31.4	16.2	16.2
14′	17.2	15.9	21.7	22.1
15′	24.9	17.2	23.8	23.4
1″	100.1	102.2	98.0	105.9
2″	83.2	76.5	75.2	76.0
3″	77.6	75.3	76.6	77.9
4″	71.9	71.9	71.6	71.6
5″	77.5	78.2	78.1	78.6
6″	62.9	68.9	70.0	69.9
1‴	105.5	110.9	104.7	104.8
2‴	76.5	78.0	75.1	75.1
3‴	77.4	80.3	77.9	77.7
4‴	71.3	75.1	71.6	71.2
5‴	78.4	66.0	78.0	78.0
6‴	62.5		62.7	62.7
1⁗				96.4
2⁗				76.8
3⁗				76.8
4⁗				71.2
5⁗				78.0
6⁗				62.7

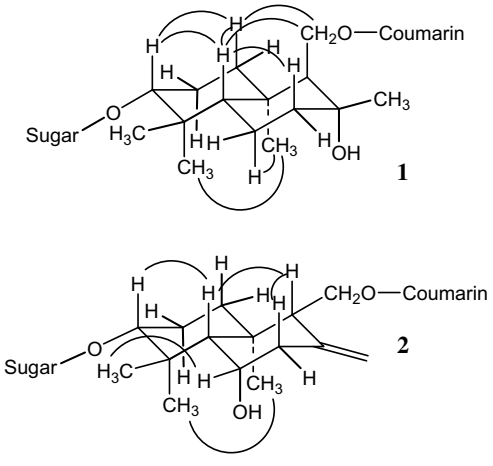

Fig. 2. Important ROESY correlations of sesquiterpene part of compounds **1** and **2**.

3.58 (*s*, H-5‴)]. The glycoside structure and its connectivity were confirmed by an HMBC experiment, which shows long-range correlations between the signals of H-3′ and C-1″, H-1″ and C-3′, H-1‴ and C-6″, and H-6″ and C-1‴. The β-configuration of the anomeric glucose unit was assigned on the basis of its typical coupling constant, $J = 8$ Hz. The proton at the anomeric carbon of apiose was determined by a ROESY experiment to posses β configuration. The ROESY experiment also supported the relative configuration of the stereogenic centres at C-3′, C-5′, C-6′, C-9′, and C-10′. In particular, ROEs H-9′/H-5′, Me-13′/Me-15′ and H-3′/H-5′ established an α-orientation for CH_2-11′, Me-13′ and Me-15′, and a β-orientation for Me-14′, H-3′, H-5′ and H-9′. The main ROE effects observed are depicted in Fig. 2. A COSY experiment also supported the proposed structure for the compound **2**. ESIMS analysis of **2** shows the positive and negative ion peaks at *m/z* 715 $[M+Na]^+$ and 727 $[M+Cl]^-$, corresponding to the molecular formula of $C_{35}H_{48}O_{14}$ for **2**, respectively. Consequently, the structure of persicaoside B (**2**), a new natural compound, was determined as shown. This is also the first report of apiose in the structure of a natural product from the genus *Ferula*.

The ^1H and ^{13}C NMR spectra (CD_3OD, Tables 1 and 2) of **3** show signals assignable to two glucopyranosyl moieties [δ 4.49 (*d*, $J = 8.0$ Hz, H-1″) and 4.33 (*d*, $J = 8.0$ Hz, H-1‴)], together with an aglycone moiety. The proton coupling constants of two glucose units were larger than 7 Hz, which is characteristic for a β-glucose. Compound **3** displayed 36 carbon signals, nine being typical of an umbelliferone skeleton, 15 signals ascribable to a sesquiterpene moiety and 12 signals concerning to two glucopyranosyl moieties. The ^1H NMR spectrum (CD_3OD, Tables 1 and 2) of **3** shows signals at δ 1.78, 1.61, 1.18 and 1.21 (all *s*, H_3-12, 13, 14, and 15), 5.46 (*t*, $J = 6.5$ Hz, H-2′) and 5.15 t (*t*, $J = 6.0$ Hz, H-6′) assignable to a linear sesquiterpene moiety. The ^{13}C NMR spectrum of **3** also displayed four signals at δ 120.4, 142.9, 125.1 and 136.5 (C-2′, 3′, 6′ and 7′) ascribable to two double bonds in the sesquiterpene region. Long-range HMBC correlations from the proton signals at δ_H 1.78 (Me-12′) to the carbon resonance at δ_C 142.9 (C-3′), δ_H 1.61 (Me-13′) to δ_C 136.5 (C-7′), and δ_H 1.18 (Me-14′) and 1.21 (Me-15′) to δ_C 81.9 (C-11′) revealed the location of the methyl groups. The glycoside structure and its connectivity were confirmed by the HMBC experiment, which show long-range correlations between the signals of H-10′ and C-1″, H-1″ and C-10′, H-1‴ and C-6″, and H-6″ and C-1‴. The configurations of the double bonds at the positions C-2′ and C-6′ were determined as E on the basis of a ROESY experiment, in which cross-peaks were observed from H-2′/H-4′, H-1′/H-12′, H-5′/H-13′ and H-6′/H-8′ pairs. ESIMS analysis of **3** shows the positive and negative ion peaks at *m/z* 747 $[M+Na]^+$ and 759 $[M+Cl]^-$, corresponding to the molecular formula of $C_{36}H_{52}O_{15}$ for **3**, respectively. Therefore, the structure of compound **3** was elucidated as 10′-*O*-[β-glucopyranosyl-(1 → 6)-β-glucopyranosyl]karatavicinol and was named

persicaoside C. The structure of persicaoside C is similar to reoselin isolated from *F. kirialovii*, *F. korshinski* and *F. pseudo-oreoselinum* (Abd El-Razek et al., 2003). In persicaoside C, the site of linkage of two glucose units to the aglycone moiety karatavicinol is C-10′ instead of C-11′ as in reoselin. On the other hand, the inter-glycosidic linkage of persicaoside C is 1 → 6 instead of 1 → 2 as in roseolin.

The ^{1}H and ^{13}C NMR spectra (CD_3OD, Tables 1 and 2) of **4** were very similar to those observed for compound **3**, containing 42 carbons and 62 protons. Comparison of the NMR spectra of compound **4** and of persicaoside C indicated that **4** exhibited anomeric signals for a third sugar at δ_H 4.61 (d, $J = 7$ Hz) and δ_C 96.4. The glycoside structure and its connectivity were confirmed by the HMBC experiment, which shows long-range correlations between the signals of H-10′ and C-1″, H-1″ and C-10′, H-1‴ and C-6″, H-6″ and C-1‴, and H-1⁗ and C-11′. ESIMS analysis of **4** shows the positive and negative ion peaks at *m/z* 909 $[M+Na]^+$ and 921 $[M+Cl]^-$, corresponding to the molecular formula of $C_{42}H_{62}O_{20}$ for **4**, respectively. Compound **4** is thus 10′-*O*-[β-glucopyranosyl-(1 → 6)-β-glucopyranosyl]11′-*O*-β-glucopyranosyl karatavicinol, namely, persicaoside D.

The isolated compounds were identified by comparing their NMR and melting point data with those previously described in the literature (Goad and Akihisa, 1997). This is the first report of sitosterol 3-*O*-β-glucoside and stigmasterol 3-*O*-β-glucoside from *F. persica*. To our knowledge, this is also the first report of sitosterol 3-*O*-β-glucoside and stigmasterol 3-*O*-β-glucoside as polar metabolites from the genus *Ferula*.

3. Experimental

3.1. General experimental procedures

Melting points were determined on an Electrothermal 9100 apparatus and are uncorrected. NMR spectra were measured on a Bruker DRX 500 (Bruker Biospin, Rheinstetten, Germany). ^{1}H NMR, ^{13}C NMR, DEPT, ^{1}H–^{1}H COSY, HMBC, HSQC, and ROESY spectra were measured using an inverse-detection probe (5 mm). The operating frequencies were 500.13 MHz for acquiring ^{1}H NMR and 125.75 MHz for ^{13}C NMR spectra. Samples were measured at 300 K in MeOH-d_4 with TMS as the internal standard. Electrospray ionization mass spectra (ESIMS) (pos. and neg. mode) were measured with a Quattro triple quadrupole mass spectrometer (VG Biotech, Altrincham, England) equipped with an ESI source. The capillary and Ispray voltages were 4 V and 5 kV, respectively. Source and capillary were heated at 60 °C and 200 °C, respectively. The mass spectrometer was operated in a conventional scanning mode using the first quadrupole. Full-scan spectra were recorded from *m/z* 100 to 1000. High-resolution time-of-flight mass spectra (HRTOFMS) were recorded on a QTOF Ultima mass spectrometer (Micromass Ltd., Manchester, UK). UV spectra were obtained from an Agilent G1315B diode array detector in MeCN–H_2O. Column chromatography was conducted with Silica gel 230–400 mesh, Merck. Preparative TLC was performed on RP-18 GF_{254s} plates (20 × 20 cm, Merck) and observation of plates was carried out under UV CAMAG spectrometer (254 nm).

3.2. Plant material

The roots of *F. persica* were collected from the Alborz Mountains, Tehran province, Iran, in May 2003. The plant was identified by the department of pharmacognosy, Faculty of Pharmacy, Tehran University of Medical Sciences. A voucher specimen (No. 6523) has been deposited at the Herbarium of the Faculty of Pharmacy (TEH).

3.3. Extraction and isolation

Dried, powdered roots of *F. persica* (250 g) were extracted with chloroform using soxhlet apparatus, then three times with methanol by maceration. The combined methanol extracts were concentrated in vacuo to give a red extract (13.1 g). Part of the extract (5.4 g) was subjected to column chromatography on silica gel (5 × 50 cm) using EtOAc–MeOH–H_2O as solvent [EtOAc 500 cm^3, EtOAc–MeOH–H_2O (24:3:0.5) 1650 cm^3, (18:3:0.5) 860 cm^3, (12:3:0.5) 1750 cm^3, (6:3:0.5) 775 cm^3 and (3:3:0.5) 650 cm^3]. The fractions were compared by TLC (silica gel using EtOAc–MeOH–H_2O as solvent), and those giving similar spots were combined. Seven fractions were finally obtained. Fraction 2 afforded 1 mg white crystals of a mixture of sitosterol 3-*O*-β-glucoside and stigmasterol 3-*O*-β-glucoside. Fraction 4 was subjected to RP-PTLC (MeOH–H_2O, 80:20) to give **1** (24.3 mg) and **2** (21.5 mg). Fraction 5 and the residue of fraction 6 were also chromatographed on RP-PTLC (MeOH–H_2O, 80:20) to yield **3** (7.3 mg) and **4** (25.3 mg), respectively.

3.4. 3′-*O*-[β-Glucopyranosyl-(1 → 2)-β-glucopyranosyl]ferukrin (*1; persicaoside A*)

White amorphous powder; UV (MeCN–H_2O) λ_{max} 225, 324 nm; ESIMS (pos. ionization mode) *m/z* 747 ($[M+Na]^+$); ESIMS (neg. ionization mode) *m/z* 759 ($[M+Cl]^-$); HRTOFMS *m/z* 725.3279 ($[M+H]^+$) (calc. for $C_{36}H_{53}O_{15}$, 725.3384); For ^{1}H and ^{13}C NMR data, see Tables 1 and 2.

3.5. 3′α-*O*-[β-Apiosyl-(1 → 6)-β-glucopyranosyl]-(5′β,9′α,10α)-8′(12′)-drimen-11′-yl-umbelliferone (*2; persicaoside B*)

White amorphous powder; UV (MeCN–H_2O) λ_{max} 225, 325 nm; ESIMS (pos. ionization mode) *m/z* 715 ($[M+Na]^+$); ESIMS (neg. ionization mode) *m/z* 727 ($[M+Cl]^-$); HRTOFMS *m/z* 693.3102 ($[M+H]^+$) (calc. for $C_{35}H_{49}O_{14}$, 693.3122); For ^{1}H and ^{13}C NMR data, see Tables 1 and 2.

3.6. 10′-O-[β-Glucopyranosyl-(1 → 6)-β-glucopyranosyl]karatavicinol (**3**; persicaoside C)

White amorphous powder; UV (MeCN–H_2O) λ_{max} 225, 325 nm; ESIMS (pos. ionization mode) *m/z* 747 ($[M+Na]^+$); ESIMS (neg. ionization mode) *m/z* 759 ($[M+Cl]^-$); HRTOFMS *m/z* 725.3289 ($[M+H]^+$) (calc. for $C_{36}H_{53}O_{15}$, 725.3384); For ^1H and ^{13}C NMR data, see Tables 1 and 2.

3.7. 10′-O-[β-Glucopyranosyl-(1 → 6)-β-glucopyranosyl]11′-O-β-glucopyranosyl karatavicinol (**4**; persicaoside D)

White amorphous powder; UV (MeCN–H_2O) λ_{max} 225, 325 nm; ESIMS (pos. ionization mode) *m/z* 909 ($[M+Na]^+$); ESIMS (neg. ionization mode) *m/z* 921 ($[M+Cl]^-$); HRTOFMS *m/z* 887.3826 ($[M+H]^+$) (calc. for $C_{42}H_{63}O_{20}$, 887.3913); For ^1H and ^{13}C NMR data, see Tables 1 and 2.

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