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Original Article

New Triterpenoids from *Peucedanum ruthenicum*

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Abstract

From hexanic extract of aerial parts of *Peucedanum ruthenicum* M. Bieb (Apiaceae) collected from Mazandaran province of Iran, four triterpenoids namely taraxasterol, y-taraxasterol, poriferasterol and b-sitosterol have been isolated by column chromatography, thin layer chromatography, and crystallization. Their structures were elucidated by spectroscopic methods (UV, IR, MS, ^{1}H and ^{13}C-NMR).

Keywords: Apiaceae; *Peucedanum ruthenicum*; Triterpenoid.
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1. Introduction

There are some 120 species of *Peucedanum* L. (Apiaceae family) widespread in Europe, Mediterranean region and south, Western and central Asia [1]. Four species of *Peucedanum* growing in Iran are: *P. glaucopruinosum* Rech., *P. knappii* Bornm., *P. translucens* KH. Rechinger [2] and *P. ruthenicum*. They are distributed in Iran [3], Russia, Europe and Turkey [4].

P. ruthenicum (Apiaceae) is a glabrous perennial plant that is distributed in the north and central part of Iran [3]. Some species of this genus have been used traditionally in treatment of colds [5], coughs due to pathogenic wind-heat, accumulation of phlegm, heat in the lung [6], anti-tussive, and are used as anti-asthmatic and as a remedy for angina [7]. Previous phytochemical studies on this species showed the presence of furanocoumarins and their glycoside derivatives, linear-type furanocoumarin glycosides and simple coumarin glycosides [8, 9]. From *P. ruthenicum*, peucedanin (furanocoumarin) and a coumarin (peuruthenicin) in the roots and rutin (flavonol glycoside) in the flowers [10] have been isolated. Several new coumarins from *P. praeruptorum* Dunn. have been reported [6]. There are some reports on the chemical analysis of voltile oil of this genus in the literature. The reported compounds of the essential oil from herb and rhizome of *P. ostruthium* were include: Sabinene (35.2%), 4-terpineol (26.6%), b-caryophyllene (16.1%) and b-humulene (15.8%) [11]. The major constituents were found to be sabinene and trans-anethole in the leaf and branch oil of *P. verticillare*. b-caryophyllene, a-phellandrene, cis-b-farnesene and b-bisabolene were found in the dried fruit oil and sabinene in the fresh fruit oil of *P. verticillare* [12].

In this research, the structures of triterpenoids from the aerial part of *P.*

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ruthenicum M.Bieb is reported.

2. Materials and methods

2.1. Plant material

The aerial parts of *P. ruthenicum* was collected in October 2004 from Roodbarak (Mazandaran province) north of Iran and was identified by H. Akhani (Dept. of Plant Biology, Faculty of Science, Tehran University, Tehran, Iran). A voucher specimen is deposited in the private herbarium of H. Akhani (Salimian 39).

2.2. General experimental procedures

Melting points were taken on a Reichert-Jung apparatus (Vienna, Austria). Ultraviolet spectra were recoded on a Shimadzu 160A spectrometer (Kyoto, Japan). Electron Ionization Mass Spectra (EIMS) were determined on a Finnigan MAT TSQ 70 (California, USA) at 70eV. ^{1}H NMR and ^{13}C NMR spectra were measured in $CDCl_3$ with tetramethylsilane (TMS) as an internal standard using a Varian 400 Unity plus spectrometer. FTIR spectra were recorded on a Nicolet 550 spectrometer (Madison , WI, USA). Column chromatography (CC) was conducted with silica gel (Kieselgel 60, 60-100 mesh ASTM; Merck, Darmstalt, Germany) and thin-layer chromatography (TLC) with Merck silica gel 60 F254 on glass plates.

2.3. Extraction procedures

Dried powdered aerial parts (100 g) of the plant were extracted with methanol by percolation. The solvent was evaporated to give a gummy residue (13.5 g). This residue when treated with 25 ml water, gave a cloudy suspension which was extracted with hexane (3×100 ml). The solvent (hexane) was evaporated and the residue (3.2 g) was chro-matographed on silica gel (petroleum ether/ EtOAc 9:1) and gave 4 fractions. The second fraction was chromatographed on PTLC (diethyl ether, toluene 1:1 saturated with HOAc 10%).

3. Results and discussion

From hexanic extract of the aerial parts of

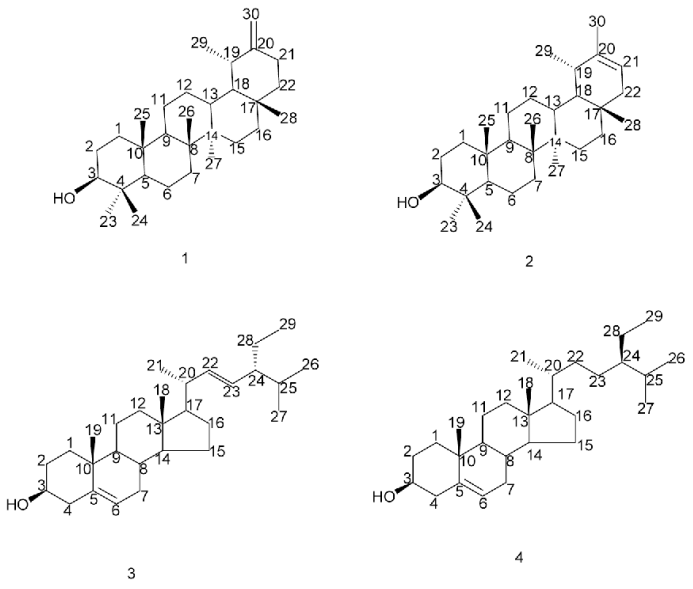

Figure 1. Structure of compounds 1-4 isolated from *Peucedanum ruthenicum* (Apiaceae).

Table 1. ^{1}H and ^{13}C-NMR data of taraxasterol, y-taraxasterol, poriferasterol and b-sitosterol.

	Taraxasterol		**y-Taraxasterol**		**Poriferasterol**		**b-Sitosterol**	
	^{13}C	^{1}H d *m*(*J*)Hz	^{13}C	^{1}H d *m*(*J*)Hz	^{13}C	^{1}H d m(*J*)Hz	^{13}C	^{1}H d *m*(*J*)Hz
C- 1	38.9	-	38.7	-	37.2	-	37.2	-
C- 2	27.3	-	27.4	-	31.7	-	31.6	-
C- 3	79.0	3.22dd6.3, 13.2	79.0	3.19m	71.8	3.52m	71.7	3.52m
C- 4	38.7	0.77(s)	38.8	-	42.3	-	42.3	-
C- 5	55.2	-	55.2	-	140.7	-	140.7	-
C- 6	18.4	-	18.3	-	121.6	5.35brd5.4	121.7	5.36d5.1
C- 7	34.0	-	34.2	-	31.9	-	31.9	-
C- 8	40.8	-	41.0	-	31.9	-	31.9	-
C- 9	50.4	-	50.3	-	50.2	-	50.2	-
C- 10	37.0	-	37.2	-	36.5	-	36.5	-
C- 11	21.5	-	21.7	-	21.1	-	21.1	-
C- 12	26.1	1.03	27.6	-	39.7	-	39.9	-
C- 13	39.2	-	29.2	-	42.3	-	42.3	-
C- 14	42.1	-	42.3	-	56.9	-	56.8	-
C- 15	26.5	0.93	27.1	-	24.4	-	24.3	-
C- 16	38.4	-	36.7	-	28.7	-	28.2	-
C- 17	34.4	-	34.3	-	56.0	-	56.1	-
C- 18	48.7	-	48.7	-	12.1	0.70(s) (3H)	11.8	0.68(s) (3H)
C- 19	39.4	-	36.3	-	19.4	1.01(s) (3H)	19.4	1.01(s) (3H)
C- 20	154.6	-	139.8	-	40.5	-	36.2	-
C- 21	25.5	-	118.9	5.26brd6.6(3H)	21.2	1.02d6.6(3H)	18.9	0.92d6.6(3H)
C- 22	38.9	-	42.1	-	138.2	5.16	33.8	-
C- 23	28.0	0.77(s) (3H)	28.1	0.84(s) (3H)	129.3	5.02	26.1	-
C- 24	15.4	0.85(s) (3H)	15.4	0.85(s) (3H)	51.2	-	45.9	-
C- 25	16.9	0.86(s) (3H)	16.2	0.86(s) (3H)	31.9	-	29.2	-
C- 26	16.0	1.02(s) (3H)	16.0	1.03(s) (3H)	21.0	0.85d6.4(3H)	19.1	0.82d6.8(3H)
C- 27	14.9	0.93(s) (3H)	14.8	0.95(s) (3H)	19.0	0.79d6.6(3H)	19.9	0.83d7.3(3H)
C- 28	19.4	0.85(s) (3H)	17.7	0.73(s) (3H)	25.4	-	23.1	-
C- 29	25.5	1.02d6.6(3H)	22.5	0.99d6.6(3H)	12.4	0.82t7.1(3H)	12.0	0.85t7.6(3H)
C- 30	107.2	4.60brs, 4.62brs	21.7	1.63brs(3H)	-	-	-	-

P. ruthenicum (Apiaceae), 4 triterpenoid fractions, (R_{f1}= 0.58, R_{f2}= 0.62, R_{f3}= 0.75 and R_{f4}= 0.78) were isolated by CC, PTLC, and crystallization. The triterpenoids, are: Taraxasterol 1, y-taraxasterol 2, poriferasterol 3 and b-sitosterol 4. The structure of these compounds were elucidated by melting point, UV, IR, MS, ^{1}H and ^{13}C-NMR spectra and by comparison of their physical data with reported in literature (Figure 1).

Compound **1** (taraxasterol): White needle crystal (4.1 mg); Mp 224-226 ºC; EIMS (70 eV) m/z (%): 426 (25), 408 (10), 229 (10), 218 (23), 207 (76), 189 (100); $C_{30}H_{50}O$; ^{1}H and ^{13}C-NMR, see Table 1 [13]. The mass spectrum of the compound 1 had $[M]^{+}$ at M/Z 426.7 suggesting the molecular formula $C_{30}H_{50}O$. The ^{1}H-NMR spectrum of the compound 1 displayed a *dd* at d 3.22 for the H-3. This is a deshielded proton as a result of OH group. The ^{1}H-NMR spectrum display methylene protons at d 4.60 and 4.62 as two singlet. The same spectrum showed signals for Me-23 at d 0.77 (s), Me-24 d 0.85 (s), Me-25 at d 0.86 (s), Me-26 at d 1.02 (s), Me-27 at d 0.93 (s), Me-28 at d 0.85 (s) and 29 at 1.02(d, J=6.6Hz). The ^{13}C-NMR spectral data (Table 1) showed 30 signals. The peaks at d 154.6 (C-20) and 107.2 (C-30) are related to olefinic carbons. The signal at d 79.0 (C-3) ppm may be related to C-O.

Compound **2** (y-taraxasterol): White needle crystal (2.6 mg); Mp 217-219ºC; EIMS (70 eV) m/z (%): 426 (16), 411 (4), 408 (4), 393 (10), 373 (5), 272 (7), 257 (7), 229 (10), 207 (69), 189 (100); $C_{30}H_{50}O$; ^{1}H and ^{13}C-NMR, see Table 1 [14].

The mass spectrum of the compound 2 had $[M]^{+}$ at M/Z 426.7 suggesting the molecular formula $C_{30}H_{50}O$.The ^{1}H- NMR spectrum of the compound 2 displayed a *m* at

d 3.19 for the H-3. This is a deshielded proton as a result of OH group. The ^{1}H-NMR spectrum displayed olefinic proton (H-21) at d 5.26 as a distorted doublet. The same spectrum showed signals for Me-23 at d 0.84 (s), Me-24 d 0.85 (s), Me-25 at d 0.86 (s), Me-26 at d 1.03 (s), Me-27 at d 0.95 (s), Me-28 at d 0.73 (s) and 29 at 0.99(d, J=6.6Hz) Me-30 at d 1.63 (brs).

The ^{13}C-NMR spectral data (Table 1) showed 30 signals. The peaks at d 139.8 (C-20) and 118.9 (C-21) are related to olefinic carbons. The signal at d 79.01 (C-3) ppm may be related to C-O.

Compound **3** (poriferasterol): White needle crystal (2.9 mg); Mp 156-158ºC; EIMS (70 eV) m/z (%): 412 (100), 397 (14), 394 (14), 379 (17), 369 (25), 351 (38), 327 (5), 314 (17), 300 (36), 283 (15), 271 (58), 255 (60), 253 (17), 241 (8), 239 (8), 231 (7), 229 (7); $C_{29}H_{48}O$; ^{1}H and ^{13}C-NMR, see Table 1 [15].

The mass spectrum of the compound 3 had $[M]^{+}$ at M/Z 412.7 suggesting the molecular formula $C_{29}H_{48}O$.The ^{1}H-NMR spectrum of the compound 3 displayed a *m* at d 3.52 for the H-3.This is a deshielded proton as a result of OH group. The ^{1}H-NMR spectrum displayed olefinic protons(H-22) at d 5.16 and (H-23) at d 5.02. The same spectrum showed signals for Me-18 at d 0.70 (s), Me-19 at d 1.01 (s), Me-21 at 1.02 (*d*, J=6.6Hz), Me-26 at d 0.85 (d, J=6.4Hz), Me-27 at d 0.79 (*d*, J=6.6Hz) and Me-29 at d 0.82 (*t*, J=7.1Hz).

The ^{13}C-NMR spectral data (Table 1) showed 29 signals. The peaks at d 138.2 (C-22) and 129.3 (C-23) are related to olefinic carbons. The signal at d 71.83 (C-3) ppm may be related to C-O.

Compound **4** (b-sitosterol): White needle crystal (7.3 mg); Mp 135-137 ºC; EIMS (70 eV) m/z (%): 414 (76), 396 (18), 381 (10), 329 (10), 255 (6), 213 (37), 199 (10), 69 (23), 57 (47), 43 (100); $C_{29}H_{50}O$; ^{1}H and ^{13}C-NMR, see Table 1 [16].

The mass spectrum of the compound 3 had $[M]^{+}$ at M/Z 414.3 suggesting the molecular formula $C_{29}H_{50}O$. The ^{1}H-NMR spectrum of the compound 4 displayed a *m* at d 3.53 for the H-3.This is a deshielded proton as a result of OH group. The ^{1}H-NMR spectrum displayed olefinic proton(H-6) at d 5.36 as adistorted doublet. The same spectrum showed signals for Me-18 at d 0.68 (*s*), Me-19 d 1.01 (*s*), Me-21at 0.92(*d*, J=6.6 Hz), Me-26 at d 0.83 (*d*, J=6.4 Hz), Me-27 at d 0.72(*d*, J=6.6 Hz) and Me-29 at d 0.84 brst.

The ^{13}C-NMR spectral data (Table 1) showed 29 signals. The peaks at d 140.77 (C-5) and 121.70 (C-6) are related to olefinic carbons. The signal at d 71.78 (C-3) ppm may be related to C-O.

4. Conclusion

This is the first report on the triterpenoids of *P. ruthenicum* M. Bieb. From hexanic extract of aerial parts of *P.ruthenicum* collected from Mazandaran province of Iran, four triterpenoids, taraxasterol, y-taraxasterol, poriferasterol and d-sitosterol have been isolated by CC, PTLC, and crystallization were characterized by melting point and spectral data.

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