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Sesquiterpene Lactone Glycosides from *Carpesium macrocephalum*

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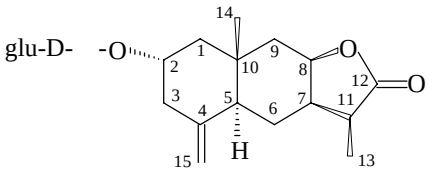
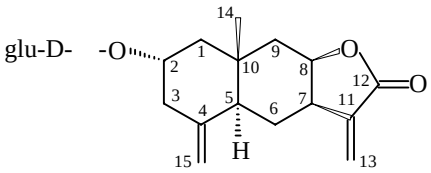
Abstract: Two new sesquiterpene lactone glycosides were isolated from the seeds of *Carpesium macrocephalum*. Their structures were elucidated as 2 -O- -D-glucopyranosy-5 , 11 H-eudesma-4 (15)-en-12, 8 -olide and 2 -O- -D-glucopyranosy-5 H-eudesma-4 (15), 11 (13)-dien-12, 8 -olide by spectral methods (HRMS, 1 D and 2 D NMR).

Keywords: *Carpesium macrocephalum*, Compositae, eudesmanolide, sesquiterpene lactone glycosides.

No previous work on the sesquiterpene lactone glycosides of the genus *Carpesium* has been found up to now. Two new sesquiterpene lactone glycosides **1** and **2** were isolated from *Carpesium macrocephalum*. Here we report the structure elucidation of them.



1



2

Compound **1**, $C_{21}H_{32}O_8$ (HRMS: revealed M+Na = 435.1996, requires 435.1989), was isolated as colorless crystal, mp: 205-207°C, [ ]$_D^{20}$ +17.9 (c 0.56, MeOH). In its ^{1}H and ^{13}C NMR spectra (**Table 1**) the typical signals for a β-D-glucopyranoside were readily recognized, which was confirmed by PC after acid hydrolysis of **1**. The remaining signals of the aglycone are similar to those of the known eudesmanoild, 2α-hydroxy-5α, 11 H-eudesma-4 (15)-en-12, 8β-olide[1]. Structural elucidation was confirmed by ^{1}H-^{1}H COSY, HMQC and HMBC spectra. The attachment of glucose to the hydroxyl at C-2 is deduced by the long range coupling between H-1′ and C-2. The large coupling constants of H-2 ($J_{2\beta,\,1\alpha}$ = $J_{2\beta,\,3\alpha}$ = 12.5Hz) confirmed the 2-hydroxy was in -orientation. Hence, the structure of the eudesmanoild glucoside was thus assigned to be **1**.

Compound **2**, $C_{21}H_{30}O_8$ (FABMS: *m/z* 433[M+Na]$^+$), was isolated as colorless

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crystal, mp: 186-188°C. Its ^{1}H and ^{13}C NMR spectra (**Table 1**) were similar to those of **1** except that the 11 -oriented methyl -lactone group (δ_C = 182.0, C; δ_H = 2.93 dq, δ_C = 42.8, CH; δ_H = 1.18 d, δ_C = 9.5, CH_3) in **1** was replaced by an -methylene- -lactone group (δ_C = 173.0, C; δ_C = 144.0, C; δ_H = 6.06, 5.70, brs, δ_C = 120.9, CH_2). So the structure of **2** was identified as 2 -O- -D-glucopyranosy-5 H-eudesma-4(15), 11(13)- dien-12, 8β-olide.

Table 1 ^{1}H, ^{13}C NMR(DEPT) and HMBC data of **1** and **2** (CD_3OD, TMS, δ_{ppm})

NO.	$^{1}H(\alpha/\beta)$ **1**	^{13}C(DEPT) **1**	HMBC(C/H) **1**	^{1}H (α/β) **2**	^{13}C (DEPT) **2**
1	1.25(br. dd, 12.5, 12.5)/1.99 (ddd, 2, 5, 12.5)	48.5 (CH2)	C-1/H-14	1.24 (t, 12, 12)/ 2.00 (ddd, 2, 5, 12)	48.4 (CH2)
2	3.87* (m)	76.0 (CH)	C-2/H-3, 1α, 1′	3.85*(m)	75.8 (CH)
3	2.11* (t, 12.5, 12.5)/ 2.79 (ddd, 2, 5, 12.5)	45.5 (CH2)	C-3/H-15	2.12* (t, 12, 12)/ 2.79 (ddd, 2, 5, 12)	45.5 (CH2)
4	-	148.4 (C)	C-4/H-3, 5	-	148.1 (C)
5	1.88 (br. d, 12.5)	46.9 (CH)	C-5/H-3β, 9β, 15, 14	1.94 (br.d, 13)	46.5 (CH)
6	1.63* (m)/1.06 (ddd, 12.5, 12.5, 12.5)	22.2 (CH2)	C-6/H-11	1.82 (ddd, 2, 6, 13)/1.26 (ddd, 13, 13, 13)	28.5 (CH2)
7	2.49 (m, 4, 7, 12.5)	41.6 (CH)	C-7/H-9β, 11, 13	3.10 (ddd, 4, 7, 13)	41.5 (CH)
8	4.52 (ddd, 2, 4, 4.5)	79.6 (CH)	C-8/H-9β	4.56 (ddd, 2, 4, 5)	78.8 (CH)
9	1.58 (dd, 4.5, 15.5)/ 2.16* (dd, 2, 15.5)	42.2 (CH2)	C-9/H-14	1.63 (dd, 5, 15.5)/ 2.20 (br. d, 15.5)	41.9 (CH2)
10	-	35.2 (C)	C-10/H-1α, 5, 9, 14	-	34.7 (C)
11	2.93 (dq, 7, 7)	42.8 (CH)	C-11/H-13	-	144.0 (C)
12	-	182.0 (C)	C-12/H-11, 13	-	173.0 (C)
13	1.18 (d, 7)	9.5 (CH3)	C-13/H-11	6.06, 5.70 (br.s)	120.9 (CH2)
14	0.78 (s)	19.1 (CH3)	C-14/H-1α	0.80 (s)	18.9 (CH3)
15	4.89, 4.62 (br.s)	109.2(CH2)	C-15/H-3	4.87, 4.59 (br.s)	109.5(CH2)
1′	4.34 (d, 8)	102.9 (CH)	C-1′/H-2′	4.34 (d, 8)	102.9 (CH)
2′	3.12 (t, 8.2)	75.1 (CH)	-	3.12 (t, 8.2)	75.1 (CH)
3′	3.34* (t, 8.8)	78.1 (CH)	C-3′/H-2′, 5′	3.34* (t, 8.8)	78.1 (CH)
4′	3.30* (t, 8.8)	71.6 (CH)	C-4′/H-3′	3.30* (t, 8.8)	71.6 (CH)
5′	3.26* (m, 2, 5.8)	77.9 (CH)	C-5′/H-4′	3.26* (m, 2, 5.8)	77.9 (CH)
6′	3.85* (dd, 2, 11.8), 3.65 (dd, 5.8, 11.8)	62.8 (CH2)	-	3.85* (dd, 2, 11.8), 3.65 (dd, 5.8, 11.8)	62.8 (CH2)

a. Signal multiplicity and coupling constants (Hz) are in parentheses;
b. *Overlapping signals.

Acknowledgment

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Reference

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