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NOTE

Isolation and Characterisation of a Sesquiterpene from *Lactuca indica*

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A new sesquiterpene lactone glycoside, named Lactuside D, was isolated from *Lactuca indica*. Spectral analysis studies suggested the compound as 3(4),11(13)-dien-eudesma-5α,6β,7αH-12,6-olide-15-O-β-D-glucopyranoside-1-β-*p*-hydroxy- phenyl-acetate.

Key Words: *Lactuca indica*, Lactuside D, Sesquiterpene lactone.

Lactuca indica belongs to compositae family, used with the whole plant or roots. It is reported that it possesses anticancer, antioxidation, antidiabetic and hypoglycemic activities [1–4]. In present study, a new sesquiterpene lactone glycoside named 3(4),11(13)-dien-eudesma-5α,6β,7α-H-12,6-olide-15-O-β-D-glucopyranoside-1-β-*p*-hydroxyphenyl-acetate (**1**) was obtained from the whole plant. In this communication, the isolation and structure elucidation of **1** has been described.

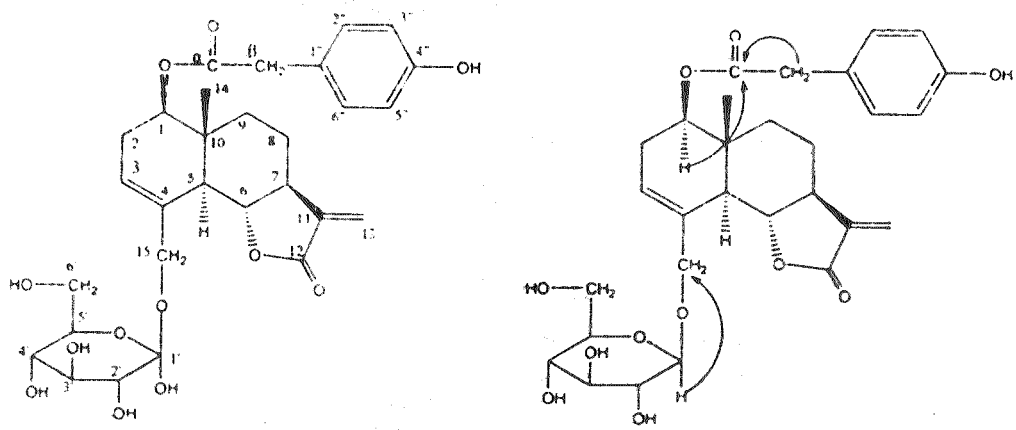

Fig. 1. Structure of compound **1** and key correlations in HMBC

An ethanol extract (70%) of *Lactuca indica* (5.0 kg) was extracted with petroleum ether, ethyl acetate *n*-butanol in turn. The ethyl acetate part (40 g) was separated by silica gel using $CHCl_3$-MeOH as eluting solvent to give six fractions.

TABLE-1

^{1}H NMR, ^{13}C NMR, DEPT DATA AND HMBC CORRELATIONS OF COMPOUND **1** (δ, ppm, TMS, C_5D_5N)

No.	^{1}H NMR	^{13}C NMR (DEPT)	HMBC
1.	5.25 (1H, m)	77.3 d	H-14
2.	2.63 (1H, m) 2.20 (1H, m)	29.8 t	H-1
3.	6.0 (1H, brs)	123.6 d	H-15
4.		134.2 s	H-15
5.	2.55 (1H, d, J = 9.9)	49.3 d	H-5, H-9, H-14
6.	4.28 (1H, m)	80.6 d	
7.	2.40 (1H, m)	50.8 d	H-8, H-13
8.	1.80 (1H, m) 1.40 (1H, m)	21.1 t	H-9
9.	1.80 (1H, m) 1.20 (1H, m)	34.4 t	H-8, H-14
10.		40.1 s	H-1, H-14
11.		140.1 s	H-13
12.		170.5 s	H-13
13.	6.18 (1H, d, J = 3.1) 5.35 (1H, d, J = 3.0)	116.2 t	
14.	1.08 (3H, brs)	12.6 q	H-1, H-9
15.	4.80 (1H, d, J = 12.8) 4.65 (1H, d, J = 12.6)	71.6 t	H-1′
Sugar moiety (GLC)			
1′	5.25 (1H, m)	104.4 d	H-2′, H-15
2′	4.10 (1H, t, J = 8.1)	75.5 d	H-3′
3′	4.28 (1H, m)	78.7 d	H-2′, H-4′
4′	4.32 (1H, m)	71.7 d	H-3′, H-6′
5′	3.95 (1H, m)	78.4 d	H-6′
6′	4.60 (1H, dd, J = 11.8, 2.3) 4.45 (1H, dd, J = 12.0, 4.8)	62.7 t	
Ester moiety			
α		171.8 s	H-1, H-β
β	3.80 (2H, brs)	41.1 t	H-2″, H-6″, H-9
1″		125.5 s	H-β, H-2″, H-6″
2″	7.5 (1H, d, J = 8.4)	131.2 d	H-β, H-3″, H-6″
3″	7.3 (1H, d, J = 8.4)	116.5 d	H-2″, H-5″
4″		158.3 s	H-2″, H-3″ H-5″, H-6″
5″	7.3 (1H, d, J = 8.4)	116.5 d	H-2″, H-6″
6″	7.5 (1H, d, J = 8.4)	131.2 d	H-β, H-2″, H-5″

Fr-6 (2.6 g) was separated by silica gel and Sephadex LH-20 to give pure **1** (50 mg) as white powder.

Compound **1**, white powder, m.p. 142–144°C, $[\alpha]_D^{20}$ + 20 (*c*0.1, methanol), HR-FAB-MS gave the m.f. $C_{29}H_{37}O_{11}$ (measured 561.2360, calcd. for $C_{29}H_{37}O_{11}$ $[M+H]^+$ 561.2336), which was supported by ^1H NMR, ^{13}C NMR and DEPT data (Table-1). Its EI-MS has two significant ion peaks at m/z 246 and 107. The IR (KBr) suggested the presence of hydroxyl (3419 cm^{-1}), α,β-unsaturated γ-lactone(1755 cm^{-1}), carbonyl (1736 cm^{-1}) and benzene ring (1616, 1600, 1516 cm^{-1}).

^1H NMR (C_5D_5N, 500 MHz) showed 4 H-signals at lower field: δ 7.5 and δ 7.3 (each 2H, d, J = 8.4 Hz) showed it is probably a *p*-disubstituted symmetrical benzene ring, which can be identified by two C-signals (δ 131.2, 116.5) appearing higher than other peaks in ^{13}C NMR. δ 6.18 and δ 5.35 (each 1H, d, J = 3.1 Hz) are suggested olefine methylene protons. Besides, the proton signal at δ 5.25 (1H, m) and the carbon signals at δ 104.4 (CH), 75.5 (CH), 78.7 (CH), 71.7 (CH), 78.4 (CH), 62.7 (CH_2) indicate the presence of a β-D-glucopyranosyloxy in **1**. ^{13}C NMR and DEPT spectra revealed 29 carbons (1 × CH_3, 7 × CH_2, 14 × CH, 7 × C) (Table-1). The signals δ 170.5, 140.1, 116.2, 80.6 confirmed the structure of α,β-unsaturated γ-lactone and the signals δ 171.8 (C), 158.3 (C), 131.2 (2 × CH), 125.5 (C), 116.5 (2 × CH), 41.1 (CH_2) can be deduced to *p*-hydroxyphenyl-acetyl, the ester moiety of this compound. By comparison with the NMR data of Ixerisoside G[5], this compound can be confirmed as a eudesmanolide-type sesquiterpene lactone glycoside. The ion peaks at m/z 107 were part of the ester moiety without carbonyl and m/z 246 was the mass of sesquiterpene skeleton without the two moiety.

In the HMBC spectrum, H-1 showed long-range correlation with Cα, while H-1′ showed correlation with C-15; thus the sugar moiety should be located at C-15 and the ester moiety located at C-1. Furthermore, H_3-14 correlated with C-1, C-5, C-9, C-10 confirmed the eudesmanolide-type sesquiterpene structure.

All naturally occurring eudesmanolide lactones have α-oriented H-7 on the basis of biosynthetic considerations[6]. It is suggested there is no NOESY correlation between H-5 and H-6, H-6 and H-7, H-14 and H-1; so H-1 and H-5 are α-oriented, while H-6 and C-14 are β-oriented. Detailed data of ^1H NMR, ^{13}C NMR, DEPT data and HMBC correlations of **1** are listed in Table-1.

From the above mentioned evidence, the chemical structure of **1** could be determined as 3(4),11(13)-dien-eudesma-5α,6β,7αH-12,6-olide-15-*O*-β-D-glucopyranoside-1-β-*p*-hydroxyphenyl-acetate, named Lactuside D[4].

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