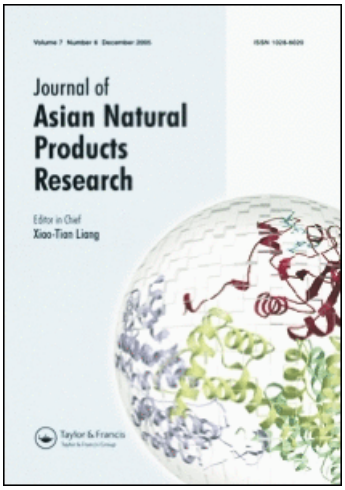

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PHENOLIC GLUCOSIDES FROM *OXYTROPIS MYRIOPHYLLA*

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Three phenolic glucosides were isolated from *Oxytropis myriophylla*. On the basis of spectral analyses, their structures were elucidated as 2-methoxy-4-(3′-hydroxy-*n*-butyl)-phenol-1-*O*-β-D-glucopyranoside (**1**), syringin (**2**), 2-methoxy-4-(3′-hydroxy-propenyl)-phenol-1-*O*-β-D-glucopyranoside (**3**). Compound (**1**) is a new phenolic glucoside named myriophylloside A, the other two compounds are isolated from this plant for the first time.

Keywords: *Oxytropis myriophylla*; Phenolic glucosides; Myriophylloside A; Spectral analyses

INTRODUCTION

Oxytropis myriophylla has been recorded in Chinese Pharmacopoeia and used for the treatment of cold and rheumatic ache. In our research, a 95% EtOH extract of the plant was separated by repeated chromatography to give three compounds. On the basis of spectral analyses, their structures were elucidated as 2-methoxy-4-(3′-hydroxy-*n*-butyl)-phenol-1-*O*-β-D-glucopyranoside (**1**), syringin (**2**) and 2-methoxy-4-(3′-hydroxy-propenyl)-phenol-1-*O*-β-D-glucopyranoside (**3**). Compound (**1**) is a new one named myriophylloside A, the other two compounds are isolated from this plant for the first time.

RESULTS AND DISCUSSION

The powdered plant was extracted with 95% EtOH, the extract was suspended in water and extracted successively with EtOAc, *n*-BuOH. The *n*-BuOH part was separated using D_{101} macroporous resin, silica gel columns, Rp-18 silica gel columns and HPLC to obtain compounds **1**–**3**.

Compound **1** was obtained as a white powder, ^{1}H NMR showed an ABX system at δ 6.97 (1H, d, *J*=8.5 Hz), 6.80 (1H, d, *J*=2.5 Hz), 6.67 (1H, dd, *J*=8.5, 2.5 Hz) and a methoxy signal at

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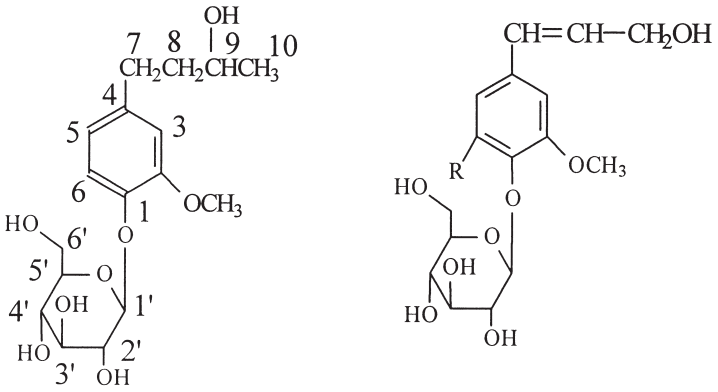

1. **2.** R=OCH_3
3. R=H

FIGURE 1 Structure of compounds **1**–**3**.

δ 3.72 (3H, s), the proton signal at δ 4.83 with J=7.5 Hz and six carbon signals at δ 100.3, 73.2, 77.0, 69.7, 76.9, 60.7 exhibited the existence of β-D-glucose. So compound **1** was characterized by ^{1}H, ^{13}C NMR as a glucoside containing a 1,2,4-trisubstituted benzene ring and a methoxy group. In $^{1}H-^{1}H$ COSY spectrum, the correlations between four proton signals at δ 1.08 (3H, d, J=6.0 Hz), 1.61 (2H, m, CH_2), 2.50, 2.60 (2H, m, CH_2) and 3.59 (1H, m, CHOH) led to a 3-hydroxy-*n*-butyl moiety. The linkage positions of three moieties (3-hydroxy-*n*-butyl moiety, a methoxy group and β-D-glucose) with the 1,2,4-trisubstituted pattern of a benzene ring were determined by HMBC spectrum, which showed the correlations between the anomeric proton signal of β-D-glucose at δ 4.83 and the aromatic carbon signal at δ 144.5, methoxy signal at δ 3.72 and the carbon signal at δ 148.8, the proton signal (δ 2.60) and three carbon signals (δ 136.2, 120.0, 112.8), which indicated the three moieties (β-D-glucosyl, methoxy group and 3-hydroxy-*n*-butyl) were linked to C-1, C-2, C-4 of the aromatic ring (see Fig. 2), respectively. All proton and carbon signals were assigned (see Table I). The

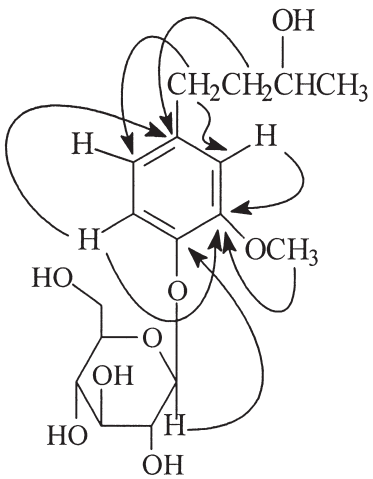

FIGURE 2 HMBC correlations for compound **1**.

HRFAB-MS of compound **1** exhibited a molecular formula $C_{17}H_{26}O_8$, a quasimolecular ion peak at 359.1702 $[M+1]^+$, and a fragment ion peak at 196.0342 $[M - glc]^+$ also confirmed the above-mentioned structure (Fig. 1). On the basis of these observations, the structure of compound **1** was established as 2-methoxy-4-(3′-hydroxy-*n*-butyl)-phenol-1-*O*-β-D-gluco-pyranoside named myriophylloside A. To our knowledge, it has not been reported previously.

EXPERIMENTAL SECTION

General Experimental Procedures

Melting points were determined on an X_4 apparatus and are uncorrected. Polarimetric data were recorded on an AA-10R Automatic Polarimeter (Optical Activity Ltd). ^{1}H NMR, ^{13}C NMR, ^{1}H–^{1}H COSY, HMQC and HMBC were recorded with a Bruker AM-500 instrument. FAB-MS was taken on AKYKY-ZHP-5# spectrometer.

Plant Material

The raw materials were collected in the city of Huerhaote, Innermongolia autonomous region, China and identified by Prof. Hubiao Chen and a specimen was deposited in a Division of Natural Medicinal Chemistry, Peking University.

Extraction and Isolation

The raw materials (7 kg) were extracted with 95% EtOH. The ethanolic extract was suspended in water and extracted with EtOAc, *n*-BuOH. The *n*-BuOH extract (110 g) was subjected to D_{101} macroporous resin eluting with water, 20, 50 and 95% EtOH. The 20% EtOH fraction (21 g) was separated by column chromatography on silica gel eluting with $CHCl_3$–MeOH–H_2O (65:35:10, lower layer) to afford four fractions. Fraction 2 was fractionated by Rp-18 silica gel column to give 15 fractions. Fractions 7–9 were purified by HPLC with MeOH–H_2O (11:89) and 1 ml/min of flow to finally yield compound **3** (7.1 mg). Fractions 11–14 were separated by preparative HPLC with MeOH–H_2O (15:85) and 8 ml/min of flow to give compound **1** (7.0 mg) and **2** (7.4 mg).

Compound **1**: white powder, mp: 177–179°C, $[\alpha]_D = +5.63$, HRFABMS *m/z* showed a molecular formula $C_{17}H_{26}O_8$ (359.1702 $[M+1]^+$, calcd 359.1698). ^{1}H NMR (500 MHz, DMSO-d): δ 6.97 (1H, d, *J*=8.5 Hz, H-6), 6.80 (1H, d, *J*=2.5 Hz, H-3), 6.67 (1H, dd, *J*=8.5, 2.5 Hz, H-5), 4.83 (1H, d, *J*=7.5 Hz, glc-H-1), 3.72 (3H, s, OCH_3), 3.59 (1H, m, H-9), 2.60

TABLE I ^{1}H, ^{13}C NMR data of compound **1** (DMSO-d_6)

No.	^{13}C	^1H	*No.*	^{13}C	^1H
1	144.5		10	21.6	1.08 (3H, d, 6.0)
2	148.8		glc 1′	100.3	4.83 (d, 7.5)
3	112.8	6.80 (d, 2.5)	2′	73.2	
4	136.2		3′	77.0	
5	120.0	6.67 (dd, 8.5, 2.5)	4′	69.7	
6	115.4	6.97 (d, 8.5)	5′	76.9	
7	31.2	2.50, 2.60 (2H, m)	6′	60.7	
8	41.00	1.61 (2H, m)	OCH_3	55.6	3.72 (s)
9	65.2	3.59 (1H, m)			

(1H, m, H-7), 2.50 (1H, m, H-7), 1.61 (2H, m, H-8), 1.08 (3H, d, *J*=6 Hz, H-10), ^{13}C NMR (DMSO-d_6): see Table I.

Compound **2**: white powder, ^{1}H NMR (300 MHz, DMSO-d_6) δ: 6.71 (2H, s, H-3,5), 6.48 (1H, d, *J*=16 Hz, H-7), 6.32 (1H, dd, *J*=16, 5 Hz, H-8), 4.94 (1H, d, *J*=7 Hz, glc-H-1), 4.83 (1H, t, *J*=5 Hz, H-9), 3.77 (6H, s, 2 × OCH_3), ^{13}C NMR (DMSO-d_6) δ: (C1-9):133.1, 153.2, 105.1, 134.5, 105.1, 153.2, 130.8, 129.0, 62.0, (C1′-6′): 103.2, 75.0, 78.0, 71.0, 77.1, 61.4, 56.9, 56.8 (2 × OCH_3). All data were identical to syringin [1].

Compound **3**: white powder, ^{1}H NMR (500 MHz, DMSO-d_6) δ: 7.05 (1H, d, *J*=2 Hz, H-3), 6.99 (1H, dd, *J*=8.5, 2 Hz, H-5), 6.89 (1H, d, *J*=8.5 Hz, H-6), 6.47 (1H, d, *J*=16 Hz, H-7), 6.29 (1H, dd, *J*=16, 5 Hz, H-8), 4.88 (1H, d, *J*=7.5 Hz, glc-H-1), 4.81 (2H, d, *J*=5 Hz, H-9), 3.77 (3 H, s, OCH_3), ^{13}C NMR (DMSO-d_6) δ: (C1-9): 146.4, 149.5, 115.7, 131.5, 119.5, 110.3, 129.4, 128.9, 62.1, (C1′-6′): 100.5, 73.7, 77.5, 70.1, 77.3, 61.1, 56.8 (OCH_3). All data were identical to [2-methoxy-4(3-hydroxy-propenyl)-phenol-1-*O*-β-D-glucoside] [2].

References

[1] Andersson, R. and Lundgren, L.N. (1988), *Phytochemistry* **27**(2), 59–562.
[2] Sticher, O. and Lahloub, F.M. (1982), *Planta Medica* **46**, 145–148.