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Three new monoterpene glucosides from *Sibiraea angustata*

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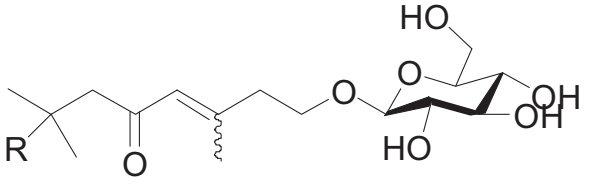
ABSTRACT

Three new monoterpene glycosides, named sibiraglycoside I(**1**), J(**2**) and K(**3**), were isolated from an aqueous extract of the aerial portion of *Sibiraea angustata*. Their structures were elucidated on the basis of extensive spectroscopic data analysis (including 1D and 2D NMR and MS experiments) and compared to literature data.



1: R= OH, *E*

2: R=OH, *Z*

3: R=H, *E*

Three new monoterpene glucosides were isolated and identified from the plant of *Sibiraea angustata*.

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Sibiraea angustata; Rosaceae; sibiraglycoside I; sibiraglycoside J; sibiraglycoside K

1. Introduction

Sibiraea angustata (Rosaceae) is a shrub, widely distributed in the western part of China, including Qinghai, Gansu, Sichuan, Xizang and Yunnan provinces, whose leaves and twig could be used as a common and civil traditional medicine in Tibet, for the treatment of indigestion and upset stomach for many years, known as 'Liucha'. And long term usage of Liucha benefits human body and reduces lipids (Institute of Botany 1985). As for, thorough chemical and bioactive studies have been performed on this plant, leading to the isolation of many active constituents, such as terpenes, phenolic acid, phenolic ester, saponins, volatile oils and polysaccharides (Yan et al. 2007; Xie et al. 2011; Wang et al. 2014). In our previous

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papers, the identification of 13 monoterpenes and monoterpene glycosides (Ito et al. 2009; Li et al. 2010, 2015; Wang et al. 2013), as well as a dicaffeic acid ester (Li et al. 2015) from the plant were reported, and the monoterpene glycosides showed significant anti-obesity activity. In our continual research, the isolation and structural elucidation of three new monoterpene glycosides, sibiraglycoside I (**1**), sibiraglycoside J (**2**) and sibiraglycoside K (**3**) from the aerial part of *S. angustat* was presented.

2. Results and discussion

The powdered residue of the aqueous extract from the aerial part of *S. angustata* was extracted by the ethanol first, and the extraction was concentrated under reduced pressure to yield a solid brown material, which was applied to a Diaion HP-20 chromatography column eluting with a gradient of C_2H_5OH/H_2O. Fortunately, three new monoterpene glycosides, sibiraglycoside I (**1**), sibiraglycoside J (**2**) and sibiraglycoside K (**3**) were obtained from the 20% Ethanol fraction by next various column chromatography (CC). Their structures were elucidated by extensive spectroscopic methods including 1D (^{1}H, ^{13}C NMR) and 2D NMR (HSQC, HMBC, ^{1}H–^{1}H COSY and ROESY) experiments as well as HRESI-MS and ESI-MS analysis and by comparison with those of the literature data.

Compound **1** was obtained as a colourless gum with an $[\alpha]_D^{20}$ value of −24.8 (*c* 0.001, CH_3OH), the molecular formula of which was determined to be $C_{16}H_{28}O_8$ from the molecular ion peak at *m/z* 371.1675 $[M + Na]^+$ (Calcd for 371.1676) in the HRESI-MS, indicating 3° of unsaturation. The IR spectrum showed absorption bands characteristic of a hydroxyl group (3388 cm^{-1}), a conjugated carbonyl group (1675 and 1611 cm^{-1}). The UV spectrum exhibited absorption maxima at 251 nm (log $\varepsilon = 2.59$). The ^{1}H NMR, ^{13}C NMR and HSQC spectra of **1** exhibited signals attributed to two methyls at δ_H 1.14 (δ_C 29.6) and δ_H 1.14 (δ_C 29.7), one methyl at δ_H 2.06 (δ_C 19.0) which may connect with an olefinic carbon according to the downfield shift of the methyl; one olefinic methine signal with a proton at δ_H 6.26 (H-4) and a carbon at δ_C 126.2 (C-4); an oxygenated methylene group with protons at δ_H 3.89, 3.60 (H-1) and carbon at δ_C 66.4 (C-1); and two methylenes with signals at δ_H 2.37 (H-2), δ_C 40.2 (C-2) and δ_H 2.50 (H-6), δ_C 56.7 (C-6). ^{13}C NMR spectrum showed an olefinic quaternary carbon at δ_C 153.4 (C-3), a carbonyl carbon at δ_C 200.0 (C-5), an oxygenated quaternary carbon at δ_C 69.0 (C-7) in addition to the above carbons.

These spectroscopic features along with two sp^2 carbon signals (one double bond) at δ_C 153.4 (C-3) and δ_C 126.2 (C-4) in the ^{13}C NMR and the ^{1}H–^{1}H COSY correlations (H-1 and H-2) suggested that **1** is an oxygenated geraniol monoterpene. The HMBC spectrum showed key long-range correlations from the following proton to carbon signals: H-1 to C-2, C-1′, C-3; H-2 to C-1, C-3, C-4, C-10; H-4 to C-2, C-3, C-5, C-10; H-6 to C-4, C-5, C-7, C-8, C-9; H-8 to C-6, C-7, C-9; H-9 to C-6, C-7, C-8; H-10 to C-2, C-3, C-4, C-5, also declaring the existence of a geraniol monoterpene. Furthermore, two methyl signals (H-8 and H-9) showed correlations to the carbon signals at C-6 and C-7, indicating two methyls were all connected to C-7.

1 contained a hexose moiety which was identified as a glucose from the methyl-1-*O*-glucoside obtained on methanolysis. The configuration of the glycosidic linkage was *β* as based on the coupling constant of the anomeric proton at δ_H 4.15 (d, $J = 7.8$ Hz). The sugar *β*-D-glucose was also identified by GC analysing the trimethylsilyl ether derivatives of its acidic hydrolysis products with standard sugars (Ito et al. 2009). In the ROESY spectrum signal at δ_H 2.37 (H-2) showed long correlations with signal at δ_H 6.26 (H-4) declaring these two

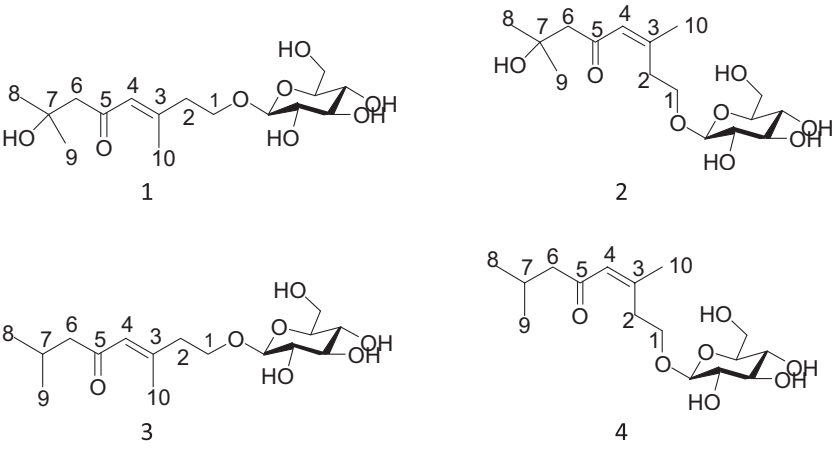

Figure 1. Structures of compounds **1**–**4**.

protons occupied the same sides, so the geometry of double bond is *E*. Thus, the structure of **1** was determined to be 3,7-dimethyl-7-hydroxy-3(*E*)-octene-5-one-1-*O*-*β*-D-glucopyranoside and named sibiraglycoside I (Figure 1).

Compound **2** was obtained as a colourless gum with an $[\alpha]_D^{20}$ value of −9.0 (*c* 0.001, CH_3OH), the molecular formula of which was determined to be $C_{16}H_{28}O_8$ from the molecular ion peak at *m/z* 371.5321 $[M + Na]^+$ (Calcd for 371.5336) in the HRESI-MS, indicating 3° of unsaturation. The IR spectrum showed absorption bands characteristic of a hydroxyl group (3397 cm^{-1}), a conjugated carbonyl group (1673 and 1610 cm^{-1}). The UV spectrum exhibited absorption maxima at 252 nm (log $\varepsilon = 2.79$). The spectral data are very similar to those of **1**. The differences are a downfield shift of the allylic methylene group [δ_H 2.78 (2H, t, H-2)] in **2** from [δ_H 2.37 (2H, t, H-2)] in **1**, and an upfield shift of the vinyl methyl group [δ_H 1.90 (3H, s, H-10)] in **2** from [δ_H 2.06 (3H, s, H-10)] in **1**. Thus, **2** was presumed to have a structure in which the double bond was isomerised to the *Z*-configuration. In fact, the appearance of a signal between H-4 and H-10 in the NOESY spectrum suggested that these protons were long correlations, so the geometry of double bond is *Z*. Thus, the structure of **2** was elucidated as 3,7-dimethyl-7-hydroxy-3(*Z*)-octene-5-one-1-*O*-*β*-D-glucopyranoside and named sibiraglycoside J (Figure 1).

Compound **3** was obtained as a colourless gum with an $[\alpha]_D^{20}$ value of −11.2 (*c* 0.001, CH_3OH), the molecular formula of which was determined to be $C_{16}H_{28}O_7$ from the molecular ion peak at *m/z* 355.2322 $[M + Na]^+$ (Calcd for 355.2317) in the HRESI-MS, indicating 3° of unsaturation. The IR spectrum showed absorption bands characteristic of a hydroxyl group (3386 cm^{-1}), a conjugated carbonyl group (1673 and 1612 cm^{-1}). The UV spectrum exhibited absorption maxima at 255 nm (log $\varepsilon = 2.43$). The spectral data are very similar to those of 3,7-dimethyl-3(*Z*)-octene-5-one-1-*O*-*β*-D-glucoside (compound **4**) (He et al. 2013). The differences are an upfield shift of the allylic methylene group [δ_H 2.37 (2H, t, H-2)] in **3** from [δ_H 2.92 (2H, m, H-2)] in **4** and a downfield shift of the vinyl methyl group [δ_H 2.07 (3H, s, H-10)] in **3** from [δ_H 1.98 (3H, s, H-10)] in **4**. Thus, **3** was presumed to have a structure in which the double bond with the *E*-configuration isomerising to compound **4** with *Z*-configuration. In addition, according to the facts that the NMR spectra data of **3** exhibited a methylene with signals at δ_H 2.37 (H-2), δ_C 40.1 (C-2) and **1** exhibited a methylene with signals at δ_H 2.37

(H-2), δ_C 40.2 (C-2), it also could be concluded that they were all trans-oriented. Thus, the structure of **3** was elucidated as 3,7-dimethyl-3(*E*)-octene-5-one-1-*O*-*β*-D-glucopyranoside and named sibiraglycoside K (Figure 1).

3. Experimental

3.1. General procedures

Optical rotations were measured on a Perkin Elmer 241 automatic digital polarimeter using Ethanol as solvent. UV spectra were recorded on a Shimadzu UV-300 spectrophotometer. IR spectra were recorded on a Nicolet 5700 FT-IR spectrometer by a transmission microscope method. NMR spectra were obtained on an INOVA-500 and Mercury-400 spectrometers in DMSO-d_6 with solvent peaks as references (δ_H 2.50, δ_C 39.7). GC was conducted using an Agilent 7890A instrument (Agilent). HRESI-MS and ESI-MS spectra were measured on an Agilent 1100 Series LC/MSD ion trap mass spectrometer. Analytical HPLC was run on a Shimadazu LC-15C instrument with a SPD-10A detector using a YMC column (RP-C_{18}, 4.6 × 250 mm, 5 μm). Preparative HPLC was performed on a Shimadzu LC-6AD instrument with an SPD-20A detector using a YMC-Pack ODS-A column (250 × 20 mm, 5 μm). The Sephadex LH-20 was obtained from Amersham Pharmacia Biotech AB Factory, Sweden. CC was performed with Diaion HP20 macroporous resin (Mitsuboshi Chemical Industries, Tokyo Japan), silica gel (60–100 and 200–300 mesh, Qingdao Marine Chemical Inc, PR China) and ODS (50 μm, YMC, Japan). TLC was carried out on glass precoated with silica gel GF254 plates (Qingdao Marine Chemical Inc, PR China). Spots were visualized under UV light or by spraying with 5% vanillin–sulphuric acid solution followed by heating.

3.2. Plant Material

S. angustata was collected in the Sichuan province, China in 2002 and identified by professor Wang Tianzhi in West China School of Pharmacy, Sichuan University, China, and a voucher specimen number specimen (001612) had been deposited in Herbarium of Jiangxi University of Traditional Chinese Medicine.

3.3. Extraction and isolation

The air-dried and powdered aerial portion of *S. angustata* (25.5 kg) was extracted with 250 L of boiling water (3 × 1 h), and the decoction was then spray-dried to yield 3.0 kg of extract. Part of the extract (1.3 kg) was extracted with ethanol under reflux (3 × 1.5 h), and the ethanol-soluble portion was concentrated under reduced pressure to give a brown solid material (500 g). This material was applied to a Diaion HP 20 chromatography column and eluted successively with a gradient of C_2H_5OH/H_2O. The 20% Ethanol fraction (40 g) was subjected to silica gel CC (800 g) and eluted with $CHCl_3/CH_3OH$ to give 13 fractions (A1–A13). Fraction A5 (1.0 g) was subjected to Sephadex LH-20 (Methanol) chromatography to give seven subfractions (A5-1–A5-7). Compound **1** (15 mg) and compound **2** (10 mg) were obtained from subfraction A5-2 (110 mg) by two rounds of continuous preparative HPLC, eluting first with 25% Methanol and second with 12% acetonitrile on a YMC-Pack ODS-A column (250 × 20 mm, 5 μm) at a flow rate of 7

mL/min and UV detection at 254 nm. Fraction A2 (5.5 g) was subjected to ODS chromatography column (50 μm, 9 g) with a CH_3OH/H_2O solvent system to give six subfractions (A2-1–A2-6). Subfraction A2-2 (1.3 g) was further fractionated to Sephadex LH-20 (Methanol) chromatography to give seven subfractions (A2-2-1–A2-2-6). Subfraction A2-2-1 (271 mg) was purified by preparative HPLC and eluted with 15% acetonitrile at a flow rate of 7 mL/min (254 nm) to obtain compound **3** (5 mg).

3.4. Acid hydrolysis and sugar analysis of compounds 1, 2 and 3

Compounds **1**–**3** (each 3 mg) were refluxed in 2 M HCl (3 mL) at 60 °C for 3 h and concentrated to give a brown material, then dissolved in water (3 mL) and partitioned by Ethyl Acetate (3 mL × 3), the aqueous layer was evaporated to dryness under reduced pressure to give the three monosaccharide residues. The absolute configuration of the monosaccharide was determined based on a reported literature (e.g. Kinjo et al. 1992). Authentic monosaccharide samples were trimethylsilylated by L-cysteine methyl ester hydrochloride and *N*-trimethylsilylimidazole. Then the derivatives were analysed by GC, and the retention times (t_R) were as follows: D-glucose at 19.83 min and L-glucose at 20.01 min. The monosaccharides obtained from the acid hydrolysis of compounds **1**–**3** were treated in a similar manner to the authentic monosaccharide samples and were both identified as D-glucose (19.83 min), which are matched to the standard D-glucose.

3.5. sibiraglycoside I (1)

Colourless gum; $[\alpha]_D^{20}$: −24.8 (*c* 0.001, CH_3OH); UV (CH_3OH) λ_{max} (log ε) 251 (2.59) nm; IRν_{max} 3388, 2972, 2928, 1675, 1611, 1375, 1203, 1159, 1079, 904, 628 cm^{-1}; ^1H NMR (DMSO-d_6, 500 MHz) δ_H: 3.89 (1H, dt, *J* = 9.8, 6.9 Hz, H-1a), 3.60 (1H, dt, *J* = 9.8, 6.8 Hz, H-1b), 2.37 (2H, t, *J* = 6.8 Hz, H-2), 6.26 (1H, s, H-4), 2.50 (2H, m, H-6), 1.14 (6H, s, H-8, 9), 2.06 (3H, s, H-10), 4.15 (1H, d, *J* = 7.8 Hz, H-1′), 2.93 (1H, td, *J* = 8.4, 5.0 Hz, H-2′), 3.12 (1H, m, H-3′, 4′), 3.03 (1H, m, H-5′), 3.67 (1H, m, H-6′a), 3.43 (1H, dt, *J* = 11.7, 5.8 Hz, H-6′b); ^{13}C NMR (DMSO-d_6, 125 MHz) δ_C: 66.4 (C-1), 40.2 (C-2), 153.4 (C-3), 126.2 (C-4), 200.0 (C-5), 56.7 (C-6), 69.0 (C-7), 29.6 (C-8), 29.7 (C-9), 19.0 (C-10), 102.8 (C-1′), 73.4 (C-2′), 76.7 (C-3′), 76.9 (C-4′), 70.1 (C-5′), 61.1 (C-6′); ESI-MS *m/z* 371 $[M + Na]^+$, 719 $[2M + Na]^+$; HRESI-MS *m/z* 348.1783 $[M]^+$ (Calcd for $C_{16}H_{28}O_8$ 348.1784).

3.6. sibiraglycoside J (2)

Colourless gum; $[\alpha]_D^{20}$: −9.0 (*c* 0.001, CH_3OH); UV (CH_3OH) λ_{max} (log ε) 252 (2.79) nm; IRν_{max} 3397, 2973, 2928, 1673, 1610, 1441, 1379, 1160, 1080, 904, 863, 720, 630 cm^{-1}; ^1H NMR (DMSO-d_6, 400 MHz) δ_H: 3.83 (1H, dd, *J* = 16.3, 7.2 Hz, H-1a), 3.54 (1H, dd, *J* = 16.5, 7.2 Hz, H-1b), 2.78 (2H, t, *J* = 7.0 Hz, H-2), 6.25 (1H, s, H-4), 2.50 (2H, m, H-6), 1.14 (6H, s, H-8, 9), 1.90 (3H, s, H-10), 4.13 (1H, d, *J* = 7.8 Hz, H-1′), 2.93 (1H, td, *J* = 8.2, 5.2 Hz, H-2′), 3.08 (1H, m, H-3′~5′), 3.65 (1H, m, H-6′a), 3.43 (1H, dt, *J* = 11.5, 5.6 Hz, H-6′b); ^{13}C NMR (DMSO-d_6, 100 MHz) δ_C: 67.1 (C-1), 33.6 (C-2), 154.4 (C-3), 126.7 (C-4), 199.7 (C-5), 56.7 (C-6), 69.1 (C-7), 29.7 (C-8), 29.7 (C-9), 25.8 (C-10), 102.8 (C-1′), 73.5 (C-2′), 76.8 (C-3′), 76.9 (C-4′), 70.0 (C-5′), 61.1 (C-6′); ESI-MS *m/z* 371 $[M + Na]^+$, 719 $[2M + Na]^+$.

3.7. sibiraglycoside K (3)

Colourless gum; $[\alpha]_D^{20}$: −11.2 (*c* 0.001, CH_3OH); UV (CH_3OH) λ_{max} (log ε) 255 (2.43) nm; IRν_{max} 3386, 2973, 2928, 1673, 1612, 1379, 1210, 1159, 1080, 904, 863, 627 cm^{-1}; ^1H NMR (DMSO-d_6, 400 MHz) δ_H: 3.89 (1H, dt, *J* = 9.4, 6.9 Hz, H-1a), 3.60 (1H, dt, *J* = 9.2, 6.8 Hz, H-1b), 2.37 (2H, t, *J* = 6.6 Hz, H-2), 6.20 (1H, s, H-4), 2.28 (2H, d, *J* = 6.9 Hz, H-6), 2.03 (1H, m, H-7), 0.86 (3H, d, *J* = 6.6 Hz, H-8), 0.85 (3H, d, *J* = 6.6 Hz, H-9), 2.07 (3H, s, H-10), 4.15 (1H, d, *J* = 7.8 Hz, H-1′), 2.93 (1H, t, *J* = 8.3 Hz, H-2′), 3.11 (1H, m, H-3′, 4′), 3.02 (1H, m, H-5′), 3.67 (1H, d, *J* = 11.7 Hz, H-6′a), 3.43 (1H, dd, *J* = 11.7, 5.7 Hz, H-6′b); ^{13}C NMR (DMSO-d_6, 100 MHz) δ_C: 66.3 (C-1), 40.1 (C-2), 153.8 (C-3), 124.6 (C-4), 200.3 (C-5), 52.7 (C-6), 24.4 (C-7), 22.4 (C-8), 22.4 (C-9), 19.0 (C-10), 102.8 (C-1′), 73.3 (C-2′), 76.8 (C-3′), 76.9 (C-4′), 70.1 (C-5′), 61.1 (C-6′); ESI-MS *m/z* 355 $[M + Na]^+$.

3.8. 3,7-dimethyl-3(Z)-octene-5-one-1-O-β-D-glucopyranoside (4)

Colourless needles; ^1H NMR (CD_3OD, 400 MHz) δ_H: 3.98 (1H, dt, *J* = 9.4, 7.2 Hz, H-1a), 3.72 (1H, m, overlapped, H-1b), 2.92 (2H, m, H=2), 6.23 (1H, s, H=4), 2.31 (2H, d, *J* = 7.0 Hz, H=6), 2.10 (1H, m, H=7), 0.92 (6H, d, *J* = 6.6 Hz, H=8, 9), 1.98 (3H, s, H=10), 4.30 (1H, d, *J* = 7.7 Hz, H=1′), 3.16 (1H, t, *J* = 8.0, 8.7 Hz, H=2′), 3.32 (1H, m, H=3′), 3.35 (1H, m, H=4′),3.28 (1H, brd, *J* = 6.4 Hz, H=5′), 3.86 (1H, d, *J* = 12.0 Hz, H=6′a), 3.68 (1H, dd, *J* = 12.0, 3.2 Hz, H=6′b); ^{13}C NMR (CD_3OD, 100 MHz) δ_C: 69.2 (C-1), 35.3 (C-2), 157.9 (C-3), 126.3 (C-4), 203.1 (C-5), 54.3 (C-6), 26.4 (C-7), 22.9 (C-8), 22.9 (C-9), 26.3 (C-10), 104.3 (C-1′), 73.0 (C-2′), 75.8 (C-3′), 76.0 (C-4′), 69.5 (C-5′), 60.7 (C-6′).

4. Conclusion

From an aqueous extract of the aerial portion of *S. angustata*, three new monoterpene glycosides named sibiraglycoside I, J and K were isolated and their structures were elucidated on the basis of the spectroscopic evidence.

Supplementary material

Supplementary material relating to this article is available online, alongside Figures S1–S2.

Acknowledgements

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Disclosure statement

No potential conflict of interest was reported by the authors.

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