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Journal of Asian Natural Products Research

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713454007>

Chemical constituents of *Stellera chamaejasme* L.

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Online publication date: 09 September 2010

To cite this Article Feng, Bao-Min , Pei, Yue-Hu and Hua, Hui-Ming(2002) 'Chemical constituents of *Stellera chamaejasme* L.', Journal of Asian Natural Products Research, 4: 4, 259 – 263

To link to this Article: DOI: 10.1080/10286020290028992

URL: <http://dx.doi.org/10.1080/10286020290028992>

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CHEMICAL CONSTITUENTS OF *STELLERA CHAMAEJASME* L.

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(Received 30 December 2001; Revised 25 February 2002; In final form 4 March 2002)

A new biflavanone, 7-methoxyneochamaejasmin A, and a new chromone derivative, isomohsenone, were isolated from the roots of *Stellera chamaejasme* L. The structures of two compounds were determined by means of ESI-MS, ^1H NMR and ^{13}C NMR, especially 2D NMR spectral analyses.

Keywords: *Stellera chamaejasme* L.; Flavonoids; Biflavonoids; Chromone derivatives

INTRODUCTION

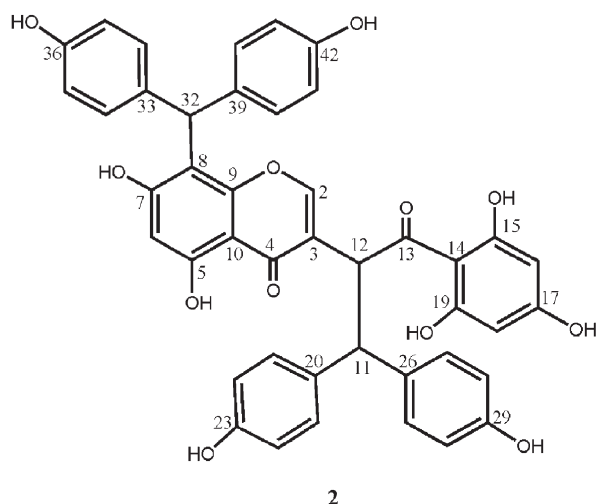
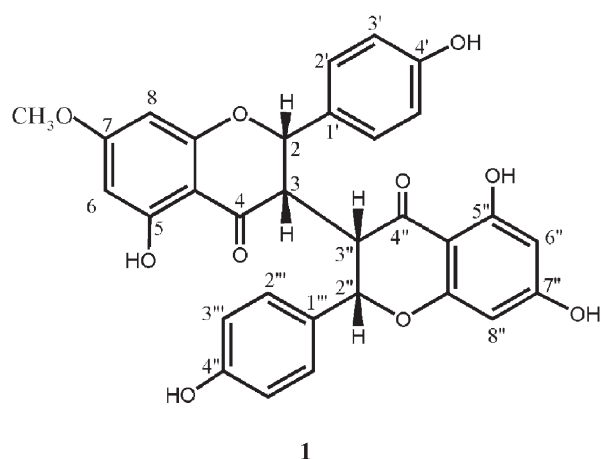
Stellera chamaejasme L. (Thymelaeaceae) is widespread in the north of China. It has been traditionally used as a herbal remedy for scabies and tinea, chronic tracheitis and tuberculosis. Recently, it has been found to possess obvious antitumor and antiviral, especially anti-HIV, activities [1–3]. *S. chamaejasme* L. has been known to contain biflavonoids and up to now a few biflavonoids have been isolated from this plant [4–7]. During our investigation of the chemical constituents of the roots of this plant, a new biflavanone, 7-methoxyneochamaejasmin A (**1**) and a new chromone derivative, isomohsenone (**2**) have been isolated Scheme 1.

RESULTS AND DISCUSSION

From the acetone-soluble parts of the EtOH extract of the roots of *S. chamaejasme* L., a new biflavanone, 7-methoxyneochamaejasmin A (**1**), and a new chromone derivative, isomohsenone (**2**) have been isolated by repeated silica gel chromatography and semi-preparative HPLC. Their structures were elucidated on the basis of spectral analysis.

Compound **1** showed positive reaction with FeCl_3 . Positive ESI-MS suggested its molecular weight was 556 ($[\text{M} + \text{H}]^+ = 557$). ^1H NMR and ^{13}C NMR signals were very similar to those of neochamaejasmin A except for the existence of the methoxy signals

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SCHEME 1 Structures of **1** and **2**.

[δ_{H} 3.79 (3H, s) and δ_{C} 56.2]. All the above information suggested that compound **1** was a methylated derivative of neochamaejasmin A. The methylated position was determined by analyzing the difference of A ring carbon signals between compound **1** and neochamaejasmin A [5], that is δ values of C-6, C-7 and C-8 shifted from δ 96.7, 168.0 and 95.8 to δ 95.4, 169.4 and 94.5, respectively. The changes of chemical shifts of C-6, C-7 and C-8 were -1.3 , $+1.4$ and -1.3 , which showed the methoxy group was located at C-7. This was further confirmed by NOESY spectrum, in which signal of the methoxy group at δ 3.79 correlated with those of 6-H and 8-H at δ 5.88 and 6.01.

Compound **2** also showed positive reaction with FeCl_3 . Its molecular weight was 740 showed by negative ESI-MS ($[\text{M} - \text{H}]^- = 739$). ^1H NMR indicated the existence of four 4-hydroxyphenyl groups [δ 7.12 (2H, d, $J = 8.2$ Hz), 6.93 (2H, d, $J = 8.1$ Hz), 6.86 (2H, d, $J = 8.1$ Hz), 6.84 (2H, d, $J = 8.1$ Hz), 6.62 (2H, d, $J = 8.1$ Hz), 6.60 (2H, d, $J = 8.1$ Hz), 6.52 (2H, d, $J = 8.2$ Hz) and 6.45 (2H, d, $J = 8.1$ Hz)], one 2,4,6-trihydroxyphenyl group [δ 5.65 (2H, s)], three isolated methines [δ 7.97 (1H, s), 6.15 (1H, s) and 5.76 (1H, s)], and a pair of coupled methines [δ 6.44 (1H, d, $J = 11.7$ Hz) and 4.50 (1H, d, $J = 11.7$ Hz)]. ^1H NMR data were very similar to those of chamaechromone [8] except for the absence of an

aromatic hydrogen and the presence of a pair of 4-hydroxyphenyl groups [δ 6.86 (2H, d, $J = 8.1$ Hz), 6.84 (2H, d, $J = 8.1$ Hz), 6.62 (2H, d, $J = 8.1$ Hz) and 6.60 (2H, d, $J = 8.1$ Hz)] and a methine proton [5.76 (1H, s)]. The two 4-hydroxyphenyl groups were proved to be connected with the methine by the fact that the signals at δ 6.86 and 6.84 showed correlations with the methine carbon at δ 42.8 in the HMBC spectrum. From all the information above, compound **2** was inferred to be a derivative of chamaechromone with a bis(4-hydroxyphenyl) methyl group located at C-8. This result was further confirmed by HMBC spectrum in which both δ 6.15 (1H, s, H-6) and 5.76 (1H, s, H-32) were correlated with δ 109.3 (C-8). The assignments of the NMR data were completed by analysis of ^1H - ^1H COSY, HMQC and HMBC spectra (Fig. 1).

EXPERIMENTAL SECTION

General Experimental Procedures

^1H NMR and ^{13}C NMR spectra were recorded on a Bruker ARX-300 spectrometer at 300 and 75 MHz, respectively. ESI-MS was taken on a LCQ LC-MS instrument. Melting points were determined on a Yanaco micro-melting points apparatus (uncorr.). Semi-preparative HPLC was carried out on an ODS column (10 \times 250 mm, phenomenex) using SPD 10AT vp HPLC meter with UV detection.

Plant Material

The roots of *S. chamaejasme* L. were collected in the west of Liaoning Province, northeast China, in September 1996 and identified by Professor Chunquan Xu of Shenyang Pharmaceutical University. A voucher specimen was deposited in the School of Traditional Chinese Medicines of Shenyang Pharmaceutical University with the No. 19960088.

Extraction and Isolation

The dry roots of *S. chamaejasme* L. (1 kg) were extracted with EtOH (95%). EtOH was evaporated and the residue was extracted with petroleum ether, ether and acetone

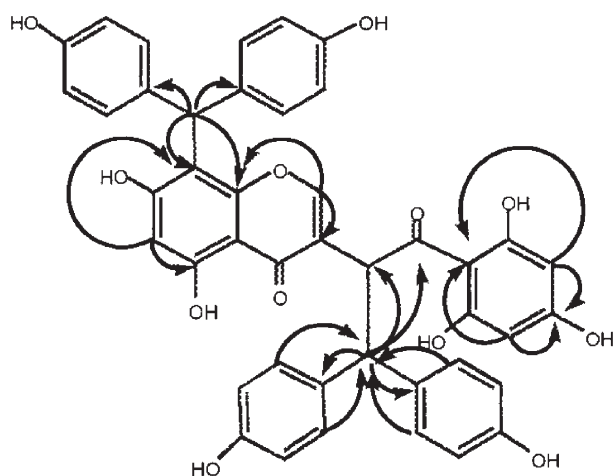


FIGURE 1 Key HMBC correlations of compound **2**.

TABLE I ^1H NMR and ^{13}C NMR data of isomohsenone (**2**)

Position	^1H NMR*	^{13}C NMR*
2	7.97 (1H, s)	155.2
3		120.0
4		179.6
5		159.6
6	6.15 (1H, s)	99.2
7		164.4
8		109.3
9		154.6
10		103.3
11	4.50 (1H, d, $J = 11.7$ Hz)	52.9
12	6.44 (1H, d, $J = 11.7$ Hz)	46.4
13		202.9
14		104.5
15		164.4
16	5.65 (1H, s)	94.8
17		165.3
18	5.65 (1H, s)	94.8
19		164.4
20 ^a		134.3
21 ^b	7.12 (1H, d, $J = 8.2$ Hz)	128.6
22 ^c	6.52 (1H, d, $J = 8.2$ Hz)	114.6
23		155.3
5-OH	12.72 (1H, s)	
24 ^c	6.52 (1H, d, $J = 8.2$ Hz)	114.6
25 ^b	7.12 (1H, d, $J = 8.2$ Hz)	128.6
26 ^a		133.4
27 ^b	6.93 (1H, d, $J = 8.1$ Hz)	129.5
28 ^c	6.45 (1H, d, $J = 8.1$ Hz)	114.7
29		155.3
30 ^c	6.45 (1H, d, $J = 8.1$ Hz)	114.7
31 ^b	6.93 (1H, d, $J = 8.1$ Hz)	129.5
32	5.76 (1H, s)	42.8
33 ^d		133.3
34 ^e	6.86 (1H, d, $J = 8.1$ Hz)	129.5
35 ^f	6.60 (1H, d, $J = 8.1$ Hz)	114.8
36		155.3
37 ^f	6.60 (1H, d, $J = 8.1$ Hz)	114.8
38 ^e	6.86 (1H, d, $J = 8.1$ Hz)	129.5
39 ^d		133.4
40 ^e	6.84 (1H, d, $J = 8.1$ Hz)	129.5
41 ^f	6.62 (1H, d, $J = 8.1$ Hz)	114.8
42		155.3
43 ^f	6.62 (1H, d, $J = 8.1$ Hz)	114.8
44 ^e	6.84 (1H, d, $J = 8.1$ Hz)	129.5

* In DMSO- d_6 .

a, b, c, d, e or f: Signals may be interchangeable.

successively. The acetone extract (20 g) was subjected to column chromatography on silica gel and eluted with CHCl_3 - CH_3OH mixture (100:1, 50:1, 30:1, 15:1, 10:1, 8:1, 5:1; v/v, about 5 l for each eluent). The fraction eluted with CHCl_3 : CH_3OH (15:1) was separated by HPLC (ODS column, 8 μM , 250 \times 10 mm, flow rate 1.5 ml/min, UV 254 nm) with H_2O : CH_3OH (3:7) as eluents to afford compound **1** (50 mg). Compound **2** (10 mg) was obtained from the CHCl_3 : CH_3OH (8:1) fraction by HPLC separation eluted with H_2O : CH_3OH (7:16).

7-Methoxyneochamaejasmin A (**1**): Pale brown powder, mp 199–201°C (MeOH- H_2O). $[\alpha]_D^{20} = +47.5$ ($c = 0.34$, MeOH). ^1H NMR (300 MHz, CD_3OD): δ 6.92 (4H, d, $J = 8.2$ Hz, H-2', 2''', 6', 6'''), 6.68 (4H, d, $J = 8.2$ Hz, H-3', 3''', 5', 5'''), 6.01 (1H, d, $J = 2.2$ Hz, H-8),

5.88 (1H, d, $J = 2.2$ Hz, H-6), 5.87 (1H, d, $J = 1.9$ Hz, H-8''), 5.72 (1H, d, $J = 1.9$ Hz, H-6''), 5.35 (1H, brs, H-2''), 5.20 (1H, brs, H-2), 3.79 (3H, s, -OCH₃), 2.96 (1H, brs, H-3''), 2.95 (1H, brs, H-3). ¹³C NMR (75 MHz, CD₃OD): δ 198.0 (C-4), 197.5 (C-4''), 169.4 (C-7), 168.5 (C-7''), 165.1 (C-5), 164.8 (C-5''), 164.5 (C-9), 164.4 (C-9''), 158.3 (C-4', 4'''), 128.3 (C-1', 1''', 2', 2''', 6', 6'''), 116.3 (C-3', 3''', 5', 5'''), 104.3 (C-10), 103.6 (C-10''), 96.8 (C-6''), 95.8 (C-8''), 95.4 (C-6), 94.5 (C-8), 82.0 (C-2''), 81.9 (C-2), 56.2 (-OCH₃), 49.9 (C-3, 3'').

Isomohsenone (**2**): brown powder, mp 228–230°C (MeOH). $[\alpha]_{\text{D}}^{20} = +124.5$ ($c = 0.15$, MeOH). ¹H NMR and ¹³C NMR data see Table I.

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