Chemical Constituents of *Impatiens pritzellii*

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【ABSTRACT】 **AIM**: To study the chemical constituents of *Impatiens pritzellii* Hook. f. var. *hupehensis* Hook. f. **METHOD**: The constituents were repeatedly separated and purified on silica column. They were identified on the basis of spectral analysis. **RESULT**: Five compounds were identified to be 2′-acetamido-3′-phenyl propyl 2-benzamido-3- phenyl propionate(**1**), spinasta-7, 22(23)-dien-3β-*O*-paltimate(**2**), 3-*O*-[6′-*O*-palmitoyl-β-*D*-glucosyl]-spinasta-7, 22(23)-diene (**3**), di (2-ethylhexyl) phthalate (**4**) and methyl docosanoate (**5**). **CONCLUSION**: All of them are isolated from Balsaminaceae for the first time.

【KEY WORDS】 *Impatiens pritzellii* Hook. f. *var. hupehensis* Hook. f.; Alkaloid; Sterol; Ester

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Impatiens pritzellii Hook. f. *var. hupehensis* Hook. f. is a balsamin- aceous plant growing in the northwestern part of Hubei Province, China. Folks use the rhizomas to treat rheumatism, diarrhea and acute bellyache[1]. By far, chemical constituents of this herb have not been reported. In our research, five compounds were separated from cyclohexane fraction. The structures of the compounds were identified by comparative analysis of their spectral, chemical and physical properties with those reported literatures.

1 Experimental

1.1 *Plant Material and Apparatus*

The rhizomas of *Impatiens pritzellii* Hook.f. *var. hupehensis* Hook.f. were purchased from Enshi city of Hubei province and identified by Prof. Dingrong Wang, Hubei Provincial Institute for Drug Control, China.

Melting points were determined on an XT4-100X micro-melting point apparatus and were uncorrected. Optical rotation was determined on a Perkin-Elmer digital polarimeter in $CHCl_3$ solution. IR spectrum was obtained on IR-460 spectrometer. ^1H NMR and ^{13}C NMR data were recorded on DRX-500 or Bruker AM-400 spectrometer, using TMS as an internal standard. MS was measured on VG Auto Spec-3000 mass spectrometer. Silica gel (100-200 mesh, Qingdao, China).

1.2 *Extraction and Isolation*

Crushed plant material (3.3 kg) was extracted three times with MeOH. The MeOH extract was filtered and concentrated under reduced pressure to give a viscous residue(1 190 g). This residue was suspended in H_2O and partitioned with cyclohexane (14.5 g), EtOAc (16 g) and n-BuOH (104.8 g) successively. The cyclohexane fraction was separated by repeated column chromatography on silica gel eluting with P.e.-EtOAc-MeOH to obtain 2′-acetamido-3′-phenyl propyl 2-benzamido-3-phenyl propionate (**1**) (26 mg), spinasta-7, 22-dien-3β-*O*-paltimate (**2**) (6 mg), 3-*O*-[6′-*O*-palmitoyl-β-*D*-glucosyl]- spinasta-7, 22(23)-diene (**3**) (64 mg), di (2-ethylhexyl) phthalate (**4**) (206 mg), methyl docosanoate (**5**) (10 mg). The structures are shown in Fig 1.

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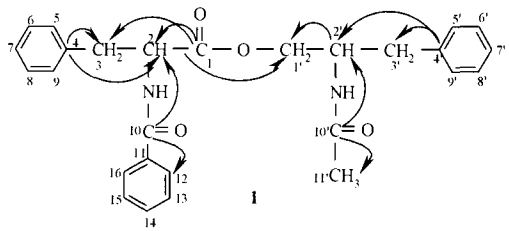

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Fig 1 Structures of compounds 1

2 Identification

2′-acetamido-3′-phenyl propyl 2-benzamido-3-phenyl propionate (1)[2], a colorless amorphous powder (Pe. + EtOAc), mp 185 ~ 186 ℃. $[\alpha]_D^{20}$ - 34° (*c* 0.5, $CHCl_3$). Its molecular formula ($C_{27}H_{28}N_2O_4$) was determined by HRFAB-MS (*m/z* 467.1946 [M + Na]$^+$, calcd. 445.2049). FAB-MS *m/z*: 445[M + H]$^+$. The positive reaction with Dragendoff's reagent was characteristic of alkaloids. IR absorption at 1726 cm^{-1} and ^{13}C NMR (δ 171.0) spectrum indicated that compound 1 has one ester carbonyl group. IR absorption at 3314, 1661, 1633 cm^{-1} and ^{13}C NMR (δ 170.8, 167.6) spectrum revealed that compound 1 had two amido groups. In ^{1}H and ^{13}C NMR spectra, compound 1 showed the presence of three single substituted benzene rings (δ 133.3, 136.3, 136.8) and a acetyl group. In addition, DEPT spectrum showed 2 tertiary carbons, 3 secondary carbons including an oxymethylene carbon at δ 64.7. From HMBC spectrum, the 3′-H(δ 2.68, CH_2) and 2′-H(δ 4.23, CH) was correlated with 4′-C(δ 136.8), 3-H(δ 3.00, CH_2) and 2-H(δ 4.68, CH) were correlated with 4-C(δ 136.3), 12, 16-H(δ 7.66, CH of phenyl) were correlated with 10-C (δ 167.6, C = O); the fragments at *m/z* 105, 91 in the FAB-MS further confirmed the linkage. The sequence of compound 1 was determined by the combination of DEPT, HSQC, HMBC, ^{1}H- ^{1}H COSY experiments, its structure was indentified as 2′-acetamido-3′-phenyl propyl 2-benzamido-3-phenyl propionate (1). Its ^{1}H NMR and ^{13}C NMR data were assigned in Table 1.

spinasta-7, 22-dien-3β-*O*-paltimate(**2**)[3]. White granular solid (EtOAc), mp 102 ~ 103℃. MF: $C_{45}H_{78}O_2$. FAB-MS *m/z*: 651[M + 1]$^+$, 607, 395, 256. IR(KBr) cm^{-1}: 2916, 2851, 1741(C = O), 1631(C = C), 970, 725. ^{1}H NMR (500 MHz, $CDCl_3$) of the paltimate moiety of 2 δ: 0.88(3H, t, CH_3), 1.25 [brs, -$(CH_2)_n$-], 1.63 (2H, m), 2.26 (2H, t, -$OCOCH_2$). ^{13}C NMR (125 MHz, $CDCl_3$) of paltimate moiety of 2 δ: 14.2(CH_3), 22.7, 24.9, 29.4-29.7, 31.9, 34.3, 173.5(C = O). ^{1}H NMR (500 MHz, $CDCl_3$) of the sterol moiety of 2 δ: 5.13-5.18 (2H, m, 7, 22-H), 5.02 (1H, dd, 23-H), 4.71 (1H, m, $W_{1/2}$ = 15.9 Hz, 3-H), 1.02 (3H, d, *J* = 6.6 Hz, 21-H), 0.79 (3H, s, 19-H), 0.55 (3H, s, 18-H). ^{13}C NMR (125 MHz, $CDCl_3$) of the spinasterol moiety of 2: 37.2, 31.9, 73.2, 34.8, 40.1, 29.7, 117.3, 139.5, 49.3, 34.4, 21.5, 39.4, 43.3, 55.1, 23.0, 28.5, 55.9, 12.0, 12.9, 40.9, 21.1, 138.1, 129.5, 51.3, 31.9, 21.5, 19.0, 25.4, 12.2.

Table 1 ^{1}H NMR and ^{13}C NMR data of compound 1

Position	δ_H	δ_C	HMBC correlations
1		170.8, s	H_2, H_3, $H_{1'}$
2	4.68(1H, dd, *J* = 7.4, 15.1)	54.7, d	H_3
3	3.00(2H, d, *J* = 7.4)	38.2, t	H_2, H_5, H_9
4		136.3, s	H_2, H_3, H_5, H_9
5	7.05(d, *J* = 7.5)	128.9, d	H_6, H_7, H_3
6	7.08(t, *J* = 7.5)	128.4, d	H_5, H_7
7	7.02(t, *J* = 7.5)	126.4, d	H_6, H_8
8	7.08(t, *J* = 7.5)	128.4, d	H_7, H_9
9	7.05(d, *J* = 7.5)	128.9, d	H_8, H_7, H_3
10		167.6, s	H_{12}, H_{16}, H_2
11		133.3, s	H_{13}, H_{15}
12	7.66(1H, d, *J* = 7.3)	127.0, d	H_{13}, H_{14}
13	7.36(1H, t, *J* = 7.3)	128.3, d	H_{12}, H_{14}
14	7.44(1H, t, *J* = 7.3)	131.8, d	H_{12}, H_{16}, H_{13}, H_{15}
15	7.36(1H, t, *J* = 7.3)	128.3, d	H_{14}, H_{16}
16	7.66(1H, t, *J* = 7.3)	127.0, d	H_{15}, H_{14}
1′	3.79(2H, qd)	64.7, t	$H_{2'}$, $H_{3'}$
2′	4.23(1H, m)	49.3, d	$H_{1'}$, $H_{3'}$
3′	2.68(2H, d, *J* = 7.3)	37.1, t	$H_{2'}$, $H_{1'}$, $H_{5'}$, $H_{9'}$
4′		136.8, s	$H_{5'}$, $H_{9'}$, $H_{3'}$, $H_{2'}$
5′	7.16(d, *J* = 7.2)	129.1, d	$H_{6'}$, $H_{7'}$, $H_{3'}$
6′	7.19(t, *J* = 7.2)	128.4, d	$H_{5'}$, $H_{7'}$
7′	7.13(t, *J* = 7.2)	126.8, d	$H_{6'}$, $H_{8'}$, $H_{5'}$, $H_{9'}$
8′	7.19(t, *J* = 7.2)	128.4, d	$H_{7'}$, $H_{9'}$
9′	7.16(d, *J* = 7.2)	129.1, d	$H_{8'}$, $H_{7'}$, $H_{3'}$
10′		171.0, s	$H_{2'}$, $H_{11'}$
11′	1.99(3H, s)	20.6, q	

3-*O*-[6′-*O*-palmitoyl-β-*D*-glucosyl]-spinasta-7, 22(23)-diene(**3**)[4-6]. White sheet crystals(EtOAc), mp 168 ~ 169 ℃. MF: $C_{51}H_{88}O_7$. IR(KBr) cm^{-1}: 2957, 2851, 1735(C = O), 1632(C = C), 1468, 1379, 1300-1150 (C-O-C), 971, 722 [-$(CH_2)_n$-, *n*

> 4]. ^{1}H NMR (500 MHz, $CDCl_3$) of the paltimate moiety of 3δ: 0.88 (3H, t, CH_3), 1.25 [brs, -$(CH_2)_n$-], 1.63 (2H, m), 2.34 (2H, t, -$OCOCH_2$). ^{13}C NMR (125 MHz, $CDCl_3$) of the paltimate moiety of 3δ: 14.1 (CH_3), 22.7, 24.9, 29.4-29.8, 31.9, 34.3, 174.4 (C = O); ^{1}H NMR (500 MHz, $CDCl_3$) of the steroid glycoside moiety of 3δ: 5.16-5.12 (2H, m, 7, 22-H), 5.03 (1H, dd, 23-H), 4.71 (1H, m, 3-H), 4.35 (1 H, d, J = 7.3Hz), 1.02 (3H, d, J = 6.6 Hz, 21-H), 0.85 (3H, d, J = 6.9Hz, 26-H) 0.80 (3H, t, J = 2.8 Hz, 29-H), 0.80 (3H, d, J = 2.8 Hz, 27-H), 0.79 (3H, s, 19-H), 0.54 (3H, s, 18-H). ^{13}C NMR (125 MHz, $CDCl_3$) of the steroid glycoside moiety of 3: 37.2, 30.1, 79.2, 34.5, 40.3, 30.1, 117.4, 139.5, 49.4, 34.5, 21.5, 39.4, 43.2, 55.1, 23.0, 28.5, 56.0, 12.0, 13.0, 40.9, 21.4, 138.1, 129.5, 51.3, 31.9, 21.1, 19.0, 25.4, 12.2, 101.2 (C-1′), 73.8 (C-2′), 76.1 (C-3′), 70.3 (C-4′), 73.8 (C-5′), 63.5 (C-6′).

Di(2-ethylhexyl) phthalate (**4**)[7]. Colorless oil. MF: $C_{24}H_{38}O_4$. FAB-MS m/z: 391[M + 1]$^+$, 279(M + 1-C_8H_{17}), 167(279-C_8H_{17} + 1), 149, 113 (C_8H_{17}). ^{1}H NMR (500 MHz, Pyr) δ:0.88 ~ 0.94(12H, m, 4 × CH_3), 1.27 ~ 1.44 (16H, m, 8, 9, 10, 12-H), 1.68 (2H, m, 7-H), 4.22 (4H, qd, 6-H), 7.53 (2H, dd, J = 6.0, 3.0 Hz), 7.71 (2H, dd, J = 6.0, 3.0 Hz). ^{13}C NMR (125 MHz, Acetone) δ: 10.9 (C-13), 14.0 (C-11), 22.9 (C-10), 23.7 (C-12), 28.8 (C-9), 30.3 (C-8), 38.7 (C-7), 68.1 (C-6), 128.8 (C-2), 130.8 (C-3), 132.4 (C-1), 167.7 (C-4).

Methyl docosanoate(**5**)[8]. White power. MF: $C_{23}H_{46}O_2$. IR(KBr) cm^{-1}:2924, 2853, 1744 (C = O), 1467. FAB-MS m/z: 355 ([M + 1]$^+$, 26), 299 (37), 271 (100), 111 (28), 97 (15), 73 (39). ^{1}H NMR (500 MHz, $CDCl_3$) δ:0.88 (3H, t, CH_3), 1.25 [brs, -$(CH_2)_n$-], 1.63 (2H, m, β-CH_2 of C = O), 2.30 (2H, t, -CH_2CO), 3.67 (3H, s, CH_3O). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 14.1, 22.7, 25.0, 29.2-29.7, 31.9, 34.1, 51.4, 173.5.

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冷水七的化学成分

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【摘 要】 **目的:** 研究凤仙花属植物冷水七 *Impatiens pritzellii* Hook. f .var. *hupehensis* Hook. f.的化学成分。**方法:** 用柱色谱分离得到化学成分,用光谱法测定其结构。**结果:** 分离鉴定了5个化合物,分别为:2′-乙酰胺基-3′-苯基苯丙醇基2-苯酰胺基-3-苯基苯丙酯(**1**),豆甾-Δ7,22-双烯-3β-棕榈酸酯(**2**),豆甾-Δ7,22-双烯-3-*O*-β-*D*-葡萄糖苷-6′-*O*-棕榈酸酯(**3**),邻苯二甲酸二(2-乙基己基)酯(**4**)和二十二烷酸甲酯(**5**)。**结论:** 这5个成分均为首次从该属植物中分得。

【关键词】 冷水七;生物碱;甾体;酯

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