

7), 103.7 (C-8), 144.1 (C-9), 113.1 (C-10); $^1\text{H-NMR}$ (Pyridine- d_5) δ : 6.25 (1H, d, $J=9.5$ Hz, H-3), 7.62 (1H, d, $J=9.5$ Hz, H-4), 7.23 (1H, s, H-5), 7.11 (1H, s, H-8)。与已知化合物的波谱数据相比较一致^[13], 确定化合物Ⅲ为 6,7-二羟基-香豆素。

化合物Ⅳ: 淡黄色针状结晶, mp 270~275 °C, 分子式为 $\text{C}_{16}\text{H}_{12}\text{O}_5$; $^{13}\text{C-NMR}$ (Pyridine- d_5) δ : 163.9 (C-2), 104.5 (C-3), 182.7 (C-4), 158.4 (C-5), 100.0 (C-6), 165.9 (C-7), 94.8 (C-8), 162.9 (C-9), 104.9 (C-10), 128.5 (C-1'), 128.5 (C-2'), 114.8 (C-3'), 163.9 (C-4'), 114.8 (C-5'), 128.5 (C-6'), 55.5 (OMe-4'); $^1\text{H-NMR}$ (Pyridine- d_5) δ : 6.90 (1H, s, H-3), 13.66 (1H, s, OH-5), 6.73 (1H, d, $J=2.0$ Hz, H-6), 6.79 (1H, d, $J=2.0$ Hz, H-8), 3.74 (3H, s, OMe-4'), 7.93 (2H, d, $J=9.0$ Hz, H-2', 6'), 7.07 (2H, d, $J=9.0$ Hz, H-3', 5'), 经与已知化合物的物理常数及波谱数据相比较一致, 确定化合物Ⅳ为 5,7-二羟基-4'-甲氧基黄酮^[14]。

化合物Ⅴ: 白色粉末, mp 269~271 °C; FAB-MS m/z : 599 $[\text{M}+\text{Na}]^+$; Molish 反应阳性, Liebermann 反应阳性。与谷甾醇葡萄糖苷对照品共薄层, R_f 值一致。确定化合物Ⅴ为谷甾醇葡萄糖苷。

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紫牡丹的化学成分研究

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摘要:目的 研究紫牡丹 *Paonia delavayi* 的化学成分。方法 利用反复硅胶柱色谱进行分离纯化, 通过理化性质和光谱数据分析鉴定化合物结构。结果 分离得到 11 个化合物, 分别鉴定为 paeonisuffral (I)、芍药苷元 (paeoniflorigenone, II)、palbinone (III)、常春藤皂苷元 (hederagenin, IV)、槲皮素-3,7-二甲氧基 (quercitrin-3,7-dimethoxy, V)、紫云英苷 (astragaloside, VI)、单棕榈酸甘油酯 (glyceryl monopalmitate, VII)、1-亚油酸-3-棕榈酸-甘油酯 (1-linoleoyl-3-palmitoylglycerol, VIII)、没食子酸甲酯 (methyl gallate, IX)、香草酸甲酯 (methyl vanillate, X)、丁香酸甲酯 (methyl syringate, XI)。结论 化合物 I, III, V~XI 为首次从该植物中分离得到。

关键词: 紫牡丹; 芍药科; 化学成分

中图分类号: R284.1

文献标识码: A

文章编号: 0253-2670(2005)05-0648-04

收稿日期: 2004-08-14

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Chemical constituents of *Paeonia delavayi*WU Shao-hua¹, WU Da-gang², CHEN You-wei¹, PENG Qian¹

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Abstract: Objective To study the chemical constituents of *Paeonia delavayi*. **Methods** Isolation and purification were carried out on repeated silica gel column chromatography. The structures of the compounds were determined by physicochemical properties and spectral analysis. **Results** Eleven compounds were isolated and identified as paeonisuffral (I), paeoniflorigenone (II), palbinone (III), hederagenin (IV), quercitrin-3, 7-dimethoxy (V), astragalin (VI), glyceryl monopalmitate (VII), 1-linoloyl-3-palmitoylglycerol (VIII), methyl gallate (IX), methyl vanillate (X), and methyl syringate (XI). **Conclusion** Compounds I, III and V—XI are isolated from this plant for the first time.

Key words: *Paeonia delavayi* Franch.; Paeoniaceae; chemical constituents

紫牡丹 *Paeonia delavayi* Franch. 又名野牡丹, 为芍药科芍药属植物, 分布于四川西南部、云南西北部及西藏东南部。其性寒、味酸辛, 根皮用于清热凉血, 治吐血、尿血、血痢、痛经等^[1], 也被用作丹皮药用, 可抑制中枢神经系统, 有扩张冠状血管, 降压、抑菌、抗炎、抗凝血等功能。据报道, 其根含有芍药苷和牡丹酚^[2]。笔者对紫牡丹的根部进行了化学成分的研究, 从其乙醇提取物中分离得到 11 个化合物, 其中化合物 I, III, V—XI 为首次从该植物中分离得到。

1 仪器与材料

熔点用四川大学科学仪器厂生产的 XRC-1 型显微熔点仪测定, 温度计未校准; UV 光谱使用日本岛津 UV-210A 仪测定; IR 光谱在 Bio-Rad FTS-135 红外光谱仪上测定; 核磁共振谱用 Bruker AM-400 型超导核磁共振仪测定, TMS 为内标; 质谱由 VG Auto Spec-3000 型质谱仪测定。薄层色谱硅胶和柱色谱硅胶均为青岛海洋化工厂出品。紫牡丹植物样品采集于云南省丽江地区, 标本由中国科学院昆明植物研究所吕正伟老师鉴定。

2 提取与分离

紫牡丹根干重 5 kg, 粉碎, 95% 工业乙醇冷浸 3 次, 滤过, 回收乙醇, 将浓缩的提取物溶于水, 用醋酸乙酯萃取 3 次, 萃取液浓缩得浸膏 130 g, 经硅胶柱层析 (200~300 目, 2.0 kg), 以氯仿-甲醇溶剂系统梯度洗脱得到 6 个组分。各组分再经反复硅胶柱色谱, 分离得到化合物 I (10 mg)、II (210 mg)、III (185 mg)、IV (32 mg)、V (23 mg)、VI (12 mg)、VII (22 mg)、VIII (37 mg)、IX (13 mg)、X (18 mg)、XI (20 mg)。

3 鉴定

化合物 I (互变异构体): 无色油状物; 分子式: $C_{10}H_{14}O_5$; 紫外无吸收; $IR_{\text{max}}^{\text{KBr}} \text{ cm}^{-1}$: 3 423, 2 943, 2 887, 1 721, 1 452, 1 353, 1 307, 1 261, 1 224, 1 187, 1 071, 1 034, 979, 834; $^1\text{H-NMR}$ (400 MHz, CD_3COCD_3) δ : 6.13 (1H, brs, OH-6b), 5.73 (1H, brs, OH-6a), 5.35 (1H, brs, OH-9b), 5.12 (1H, d, $J=2.7$ Hz, H-9a), 4.90 (1H, brs, OH-3a), 3.86 (1H, dd, $J=8.3, 5.4$ Hz, H-8a), 3.56 (1H, d, $J=8.3$ Hz, H-8'a), 3.44 (1H, dd, $J=11.2, 5.1$ Hz, H-8b), 3.27 (1H, dd, $J=11.2, 8.7$ Hz, H-8'b), 2.71 (1H, d, $J=17.2$ Hz, H-2b), 2.63 (1H, m, H-4b), 2.42 (1H, d, $J=17.2$ Hz, H-2'b), 2.41 (1H, m, H-4a), 2.29 (1H, m, H-7a), 2.27 (1H, dd, $J=14.0, 2.9$ Hz, H-5b), 2.24 (1H, dd, $J=14.0, 2.5$ Hz, H-5'b), 2.09 (1H, dd, $J=14.0, 4.4$ Hz, H-5a), 2.03 (1H, m, H-7), 1.96 (1H, d, $J=14.1$ Hz, H-2a), 1.85 (1H, dd, $J=14.0, 1.0$ Hz, H-5'a), 1.76 (1H, d, $J=14.1$ Hz, H-2'a), 1.20 (3H, s, H-10b), 1.11 (3H, s, H-10a); $^{13}\text{C-NMR}$ (100 MHz, CD_3COCD_3) δ : 79.1 (s, C-1a, 1b), 45.4 (t, C-2a), 47.7 (t, C-2b), 107.2 (s, C-3a), 210.7 (s, C-3b), 45.6 (d, C-4a), 47.4 (d, C-4b), 32.1 (t, C-5a), 35.6 (t, C-5b), 102.4 (s, C-6a, 6b), 43.6 (d, C-7a), 47.0 (d, C-7b), 66.5 (t, C-8a), 60.9 (t, C-8b), 100.5 (d, C-9a), 100.4 (d, C-9b), 22.6 (q, C-10a), 21.9 (q, C-10b); EI-MS (70 eV) m/z (%): 214 [$\text{M}]^+$ (14), 196 [$\text{M}-\text{H}_2\text{O}]^+$ (60), 150 (74), 140 (14), 122 (53), 114 (30), 97 (66), 85 (69), 69 (80), 55 (100)。依据光谱分析, 确定化合物 I 为 paeonisuffral^[3]。

化合物 II: 无色油状物; 分子式: $\text{C}_{17}\text{H}_{18}\text{O}_6$; UV

$\lambda_{\max}^{\text{MeOH}}$ nm: 240.5, 274.5, 282.0; $\text{IR}_{\max}^{\text{KBr}}$ cm^{-1} : 3 421, 2 971, 1 723, 1 600, 1 451, 1 397, 1 315, 1 278, 1 102, 1 171, 1 033, 963, 712; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ : 7.98 (2H, d, $J=7.8$ Hz, H-2', 6'), 7.54 (1H, t, $J=7.8$ Hz, H-4'), 7.39 (2H, t, $J=7.8$ Hz, H-3', 5'), 5.45 (1H, s, H-9), 4.34 (1H, dd, $J=11.5, 5.6$ Hz, H-8a), 4.04 (1H, dd, $J=11.5, 9.5$ Hz, H-8b), 2.87 (1H, m, H-4), 2.65 (2H, d, $J=2.4$ Hz, H-2), 2.38 (1H, dd, $J=13.5, 3.5$ Hz, H-5 α), 2.36 (1H, m, overlap, H-7), 2.18 (1H, dd, $J=13.5, 2.4$ Hz, H-5 β), 1.29 (3H, s, H-10); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ : 78.6 (s, C-1), 46.6 (t, C-2), 209.6 (s, C-3), 46.3 (d, C-4), 34.2 (t, C-5), 101.6 (s, C-6), 43.0 (d, C-7), 62.6 (t, C-8), 99.7 (d, C-9), 21.1 (q, C-10), 129.7 (s, C-1'), 129.6 (d, C-2', 6'), 128.4 (d, C-3', 5'), 133.2 (d, C-4'), 166.1 (s, C-7'); EI-MS (70 eV) m/z (%): 318 [$\text{M}]^+$ (1), 196 [$\text{M} - \text{benzoic acid}]^+$ (54), 178 (9), 150 (66), 122 (43), 105 (100), 77 (59), 69 (44), 55 (33)。依据光谱分析, 确定化合物 II 为芍药苷元^[3]。

化合物 III: 红色针状结晶 (石油醚-丙酮); mp 191~192 °C; 分子式: $\text{C}_{22}\text{H}_{30}\text{O}_4$; UV $\lambda_{\max}^{\text{MeOH}}$ nm: 243, 386.5; $\text{IR}_{\max}^{\text{KBr}}$ cm^{-1} : 3 382, 2 943, 2 865, 1 751, 1 688, 1 590, 1 430, 1 386, 1 280, 1 168, 1 073, 1 037, 987; $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ : 6.85 (1H, dd, $J=10.1, 3.4$ Hz, H-12), 6.40 (1H, dd, $J=10.1, 2.4$ Hz, H-11), 3.27 (1H, dd, $J=11.6, 4.8$ Hz, H-3), 2.04 (1H, d, $J=4.0$ Hz, H-9), 2.01 (1H, m, H-7 β), 1.93 (1H, dt, $J=11.2, 3.5$ Hz, H-1 α), 1.73 (1H, m, H-2 α), 1.70 (1H, m, H-6 α), 1.68 (1H, m, H-7 α), 1.64 (1H, m, H-2 β), 1.50 (1H, m, H-6 β), 1.18 (3H, s, H-30), 1.10 (1H, td, $J=11.2, 4.1$ Hz, H-1 β), 1.00 (3H, s, H-28), 0.90 (3H, s, H-19), 0.86 (1H, dd, $J=12.0, 2.0$ Hz, H-5), 0.81 (3H, s, H-18), 0.78 (3H, s, H-29); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ : 38.2 (t, C-1), 26.8 (t, C-2), 78.6 (d, C-3), 39.0 (s, C-4), 55.0 (d, C-5), 18.0 (t, C-6), 33.4 (t, C-7), 40.1 (s, C-8), 56.1 (d, C-9), 37.3 (s, C-10), 141.5 (d, C-11), 120.2 (d, C-12), 146.4 (s, C-13), 50.8 (s, C-14), 201.2 (s, C-15), 180.9 (s, C-16), 151.1 (s, C-17), 19.0 (q, C-18), 18.3 (q, C-19), 27.8 (q, C-28), 15.0 (q, C-29), 19.5 (q, C-30); EI-MS (70 eV) m/z (%): 358 [$\text{M}]^+$ (43), 340 [$\text{M} - \text{H}_2\text{O}]^+$

(44), 325 (10), 297 (7), 231 (8), 220 (19), 207 (46), 189 (100), 173 (35), 133 (40), 119 (49), 105 (54), 91 (57); HREI-MS m/z : 358.214 5 [$\text{M}]^+$ (calcd for $\text{C}_{22}\text{H}_{30}\text{O}_4$, 358.214 4)。依据光谱分析, 确定化合物 III 为 palbinone^[4]。

化合物 IV: 白色无定形粉末; mp 214~315 °C; 分子式: $\text{C}_{30}\text{H}_{48}\text{O}_4$; UV $\lambda_{\max}^{\text{MeOH}}$ (log ϵ): 206.0 (3.78) nm; $\text{IR}_{\max}^{\text{KBr}}$ cm^{-1} : 3 449, 2 943, 2 577, 1 697, 1 463, 1 386, 1 267, 1 206, 1 037, 1 013, 653; $^1\text{H-NMR}$ (400 MHz, $\text{C}_5\text{D}_5\text{N}$) δ : 5.50 (1H, brs, H-12), 4.17 (1H, d, $J=10.3$ Hz, H-23a), 3.71 (1H, d, $J=10.3$ Hz, H-23b), 3.28 (1H, dd, $J=13.7, 4.0$ Hz, H-3 α), 1.25, 1.06, 1.03, 1.01, 0.98, 0.94 (18H, s, $\text{CH}_3 \times 6$); $^{13}\text{C-NMR}$ (100 MHz, $\text{C}_5\text{D}_5\text{N}$) δ : 38.9 (t, C-1), 28.4 (t, C-2), 73.9 (d, C-3), 42.9 (s, C-4), 49.0 (d, C-5), 18.7 (t, C-6), 33.1 (t, C-7), 39.9 (s, C-8), 48.3 (d, C-9), 37.3 (s, C-10), 23.8 (t, C-11), 122.7 (d, C-12), 144.9 (s, C-13), 42.3 (s, C-14), 28.4 (t, C-15), 23.9 (t, C-16), 46.6 (s, C-17), 46.8 (d, C-18), 42.1 (t, C-19), 31.0 (s, C-20), 34.3 (t, C-21), 33.3 (t, C-22), 68.5 (t, C-23), 13.0 (q, C-24), 16.0 (q, C-25), 17.6 (q, C-26), 26.2 (q, C-27), 180.1 (s, C-28), 33.3 (q, C-29), 23.8 (q, C-30); EI-MS (70 eV) m/z (%): 472 [$\text{M}]^+$ (4), 454 [$\text{M} - \text{H}_2\text{O}]^+$ (3), 426 [$\text{M} - \text{HCOOH}]^+$ (5), 248 (100), 223 (11), 203 (75), 187 (15), 175 (21), 119 (20), 107 (17), 95 (19), 81 (21), 69 (21)。依据光谱分析, 确定化合物 IV 为常春藤皂苷元^[5]。

化合物 V: 黄色粉末; mp 238~239 °C; 分子式: $\text{C}_{17}\text{H}_{14}\text{O}_7$; UV $\lambda_{\max}^{\text{MeOH}}$ nm: 255.5, 272.0, 341.5; $\text{IR}_{\max}^{\text{KBr}}$ cm^{-1} : 3 420, 1 680, 1 631, 1 168, 1 032; $^1\text{H-NMR}$ (400 MHz, DMSO-d_6) δ : 7.55 (1H, d, $J=2.0$ Hz, H-2'), 7.48 (1H, dd, $J=8.5, 2.0$ Hz, H-6'), 6.87 (1H, d, $J=8.5$ Hz, H-5'), 6.70 (1H, d, $J=2.0$ Hz, H-6), 6.32 (1H, d, $J=2.0$ Hz, H-8), 3.81 (3H, s, OCH_3), 3.77 (3H, s, OCH_3); $^{13}\text{C-NMR}$ (100 MHz, DMSO-d_6) δ : 155.2 (s, C-2), 136.4 (s, C-3), 176.9 (s, C-4), 159.3 (s, C-5), 97.2 (d, C-6), 164.8 (s, C-7), 92.3 (d, C-8), 155.5 (s, C-9), 105.3 (s, C-10), 120.3 (s, C-1'), 115.7 (d, C-2'), 144.7 (s, C-3'), 148.0 (s, C-4'), 115.9 (d, C-5'), 119.6 (d, C-6'), 58.9 (q, OCH_3), 56.5 (q, OCH_3); EI-MS (70 eV) m/z (%): 330 [$\text{M}]^+$ (100), 315 [$\text{M} - \text{CH}_3$]⁺ (31), 300

(4), 287(5), 203(4), 160(4), 103(9), 60(19)。依据光谱分析, 确定化合物 V 为槲皮素-3, 7-二甲氧基^[6]。

化合物 VI: 黄色粉末; mp 209~211 °C; 分子式: $C_{21}H_{20}O_{11}$; 依据 UV, IR, 1H -NMR, ^{13}C -NMR 光谱分析, 确定化合物 VI 为紫云英苷^[7]。

化合物 VII: 白色粉末(甲醇); mp 69~70 °C; 分子式: $C_{19}H_{38}O_4$; 1H -NMR (400 MHz, $CDCl_3$) δ : 4.21 (1H, dd, $J=6.0, 11.6$ Hz, H-1a), 4.12 (1H, dd, $J=4.7, 11.6$ Hz, H-1b), 3.94 (1H, m, H-2), 3.68 (1H, dd, $J=5.8, 11.2$ Hz, H-3a), 3.62 (1H, dd, $J=4.0, 11.2$ Hz, H-3b), 2.77 (1H, brs, OH), 2.35 (2H, t, $J=7.6$ Hz, H-2'), 1.62 (2H, m, H-15'), 1.25 (24H), 0.88 (3H, t, $J=7.5$ Hz, H-16'); ^{13}C -NMR (100 MHz, $CDCl_3$) δ : 65.2 (t, C-1), 70.4 (d, C-2), 63.4 (t, C-3), 174.4 (s, C-1'), 34.2 (t, C-2'), 31.9 (t, C-3'), 29.7-29.1 (t, C-4' to C-13'), 24.9 (t, C-14'), 22.7 (t, C-15'), 14.0 (q, C-16'); EI-MS (70 eV) m/z (%): 330 [M]⁺ (3), 313 (4), 299 (34), 270 (20), 257 (34), 239 (79), 134 (78), 112 (42), 98 (100), 84 (69), 74 (74), 57 (96)。依据光谱分析, 确定化合物 VII 为单棕榈酸甘油酯^[8]。

化合物 VIII: 无色油状物; 分子式: $C_{37}H_{68}O_5$; 1H -NMR (400 MHz, $CDCl_3$) δ : 5.33 (4H, m, H-9', 10', 12', 13'), 4.29 (2H, dd, $J=4.3, 11.9$ Hz, H-1), 4.13 (2H, dd, $J=6.0, 11.9$ Hz, H-3), 2.74 (1H, m, H-2), 2.30 (4H, m, H-2', 2''), 0.84 (6H, m, H-18', 16''); ^{13}C -NMR (100 MHz, $CDCl_3$) δ : 62.1 (t, C-1, 3), 69.0 (d, C-2), 172.8 (s, C-1'), 173.2 (s, C-1''), 130.2 (d, C-9'), 130.0 (d, C-10'), 128.1 (d, C-12'), 128.0 (d, C-13'), 34.2-22.7 (t, CH_2), 14.0 (q, C-18', 16''); negative FAB-MS m/z (%): 591 [$M-1$]⁻ (5), 279 [$C_{17}H_{31}COO$]⁻ (100), 255 [$C_{15}H_{31}COO$]⁻ (98)。依据光谱分析, 确定化合物 VIII 为 1-亚油酸-3-棕榈酸-甘油酯。

化合物 IX: 白色无定形粉末; mp 182~183 °C; 分子式: $C_8H_8O_5$; UV, IR, 1H -NMR, ^{13}C -NMR 和 EI-MS 数据与文献^[9]对照一致, 确定该化合物为没食

子酸甲酯。

化合物 X: 白色无定形粉末; mp 131~132 °C; 分子式: $C_9H_{10}O_4$; UV, IR, 1H -NMR, ^{13}C -NMR 和 EI-MS 数据与文献^[10]对照一致, 确定该化合物为香草酸甲酯。

化合物 XI: 白色无定形粉末; mp 125~126 °C; 分子式: $C_{10}H_{12}O_5$; UV, IR, 1H -NMR, ^{13}C -NMR 和 EI-MS 数据与文献^[11]对照一致, 确定该化合物为丁香酸甲酯。

致谢: 中国科学院昆明植物研究所植物化学与西部植物资源持续利用国家重点实验室仪器组测定所有光谱数据。

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