

· 基础研究 ·

银线草化学成分研究[△]王传琦^{1,2}, 金新宇¹, 韩悦¹, 刘心雨¹, 柳莹¹, 庄鹏宇^{1*}

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[摘要] **目的:** 对金粟兰属植物银线草 *Chloranthus japonicus* Sieb. 全草的化学成分进行研究, 以探究其药理活性物质基础。**方法:** 采用硅胶、凝胶 Sephadex LH-20、半自动制备液相等色谱方法对银线草的乙酸乙酯部位进行分离纯化, 通过 1D-NMR、紫外(UV)光谱、质谱(MS)技术对化合物的结构进行鉴定。**结果:** 从银线草 75% 乙醇提取物的乙酸乙酯部位共分离得到 9 个化合物, 分别鉴定为金合欢素(1)、千层纸素 A(2)、7E-3, 4-二氧甲基苯并[2-苯基-胸苷](3)、落叶松脂素(4)、9 α -羟基紫菀内酯(5)、银线草醇 B(6)、金粟兰素 C(7)、白术内酯 III(8)、白术内酯 II(9)。**结论:** 化合物 1~4 为首次在金粟兰属中分离得到。

[关键词] 银线草; 金粟兰属; 金合欢素; 千层纸素 A; 7E-3, 4-二氧甲基苯并[2-苯基-胸苷]; 落叶松脂素

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Study on Chemical Constituents of *Chloranthus japonicus*WANG Chuan-qi^{1,2}, JIN Xin-yu¹, HAN Yue¹, LIU Xin-yu¹, LIU Ying¹, ZHUANG Peng-yu^{1*}

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[Abstract] **Objective:** To study chemical constituents of the whole plant of *Chloranthus japonicus* Sieb. and explore the basis of pharmacological substances. **Methods:** The compounds were isolated and purified by repeated silica gel, gel Sephadex LH-20 and semi-automatic preparative liquid chromatography from EtOAc extraction of *C. japonicus*. The structure of the compound was identified by 1D-NMR, UV and MS. **Results:** Nine compounds were isolated, which were identified as acacetin (1), 6-methoxybaicalein (2), 7E-3, 4-dioxymethylzimsaure-[2-phenyle-thylamid] (3), lariciresinol (4), 9 α -hydroxyasterolide (5), shizukaol B (6), spicachlorantin C (7), atractylenolide III (8), atractylenolide II (9). **Conclusion:** Compounds 1-4 were isolated from japonicus for the first time.

[Keywords] *Chloranthus japonicus* Sieb.; *Chloranthus*; acacetin; 6-methoxybaicalein; 7E-3, 4-dioxymethylzimsaure-[2-phenyle-thylamid]; lariciresinol

银线草 *Chloranthus japonicus* Sieb. 又名灯笼花、分叶芹、假细辛, 为金粟兰科金粟兰属多年生草本植物。银线草产于我国吉林、辽宁、河北、山东、山西、陕西、甘肃, 亦分布于俄罗斯远东地区、朝鲜、日本。其生于林下阴湿处, 根状茎可提取芳香油, 全株供药用, 具有祛湿散寒、活血止痛、散瘀解毒等功效, 用于治疗风寒咳嗽、跌打损伤、痈肿

疮疖、妇女经闭等^[1]。研究表明, 银线草主要含有倍半萜、倍半萜多聚体和单萜类化学成分^[2]。现代药理研究表明, 银线草提取物具有抗菌、抗人免疫缺陷病毒(HIV)和抗炎等生物活性^[3]。本研究对银线草化学成分进行分离与鉴定, 从银线草 75% 乙醇提取物的乙酸乙酯部位共分离得到 9 个化合物, 分别鉴定为金合欢素(1, acacetin)、千层纸素 A

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(**2**, 6-methoxybaicalein)、7E-3, 4-二氧甲基苯并[2-苯基-胸苷] (**3**, 7E-3, 4-dioxymethylenzimtsaure-[2-phenyle-thylamid])、落叶松脂素(**4**, lariciresinol)、9 α -羟基紫菀内酯(**5**, 9 α -hydroxyasterolide)、银线草醇B(**6**, shizukaol B)、金粟兰素C(**7**, spicachlorantin C)、白术内酯Ⅲ(**8**, atractylenolide Ⅲ)、白术内酯Ⅱ(**9**, atractylenolide Ⅱ)。其中, 化合物**1**~**4**为首次在金粟兰属中分离得到。

1 材料

1.1 试药

银线草的全草采自陕西省安康市宁陕县, 经华北理工大学药学院庄鹏宇副教授鉴定为 *Chloranthus japonicus* Sieb., 样品留样保存于华北理工大学药学院天然药物研究室(标本号: ID2016-1)。

硅藻土(烟台化学研究所); 75%乙醇、无水乙醇、石油醚、乙酸乙酯、二氯甲烷、甲醇、乙腈(天津天力化学试剂公司); 纯净水(杭州娃哈哈集团有限公司)。

1.2 仪器

JASCO-V650型紫外分析仪(北京金先锋光电科技有限公司); 1260型分析型高效液相色谱仪(美国Agilent公司); 6AT型半自动制备型高效液相色谱仪(日本岛津公司); Avance II HD 600型核磁共振仪(德国Bruker公司); 柱色谱硅胶400~500目、薄层色谱硅胶H(青岛海洋化工有限公司)。

2 提取与分离

银线草全草(4 kg)干燥粉碎, 用75%乙醇水回流提取(8倍量溶剂, 回流4 h, 2次), 40℃减压干燥得到48 g浸膏。浸膏用甲醇复溶吸附于等量的硅藻土上并完全干燥, 依次用石油醚、乙酸乙酯进行洗脱(4次), 分别得到石油醚洗脱部分720 g和乙酸乙酯洗脱部分700 g。取乙酸乙酯洗脱部分进行硅胶柱色谱分离, 洗脱剂为二氯甲烷-甲醇, 梯度洗脱(100:0→0:100), 根据薄层色谱检测结果进行相似成分合并, 得到YEA~YEF 5个组分。YEA经过凝胶柱色谱纯化, 甲醇-水(30:70)洗脱, 得到化合物**1**(12.5 mg)和**2**(6.1 mg); YEB经过硅胶柱色谱(石油醚-乙酸乙酯, 10:1)洗脱, 得到化合物**3**(5.2 mg)和**4**(7.6 mg); YEC经过硅胶柱

色谱(石油醚-乙酸乙酯, 5:1)洗脱, 得到化合物**5**(20.6 mg)和**6**(40.8 mg); YED经过凝胶柱色谱(二氯甲烷-石油醚-甲醇, 4:4:1)洗脱, 得到化合物**7**(25.6 mg)。化合物**7**母液经半自动制备液相纯化(乙腈-水, 45:55)得到化合物**8**(5.2 mg)和**9**(2.3 mg)。

3 结果

化合物**1**: 黄色粉末, ESI-MS m/z 285 $[M+H]^+$, 提示分子式为 $C_{16}H_{12}O_5$; 1H -NMR(600 MHz, $CDCl_3$) δ : 7.28(1H, s, H-3), 6.47(1H, s, H-6), 6.72(1H, s, H-8), 7.58(2H, d, $J=7.3$ Hz, H-2', 6'), 7.94(2H, d, $J=7.3$ Hz, H-13, 15), 3.91(3H, s, OMe); ^{13}C -NMR(150 MHz, $CDCl_3$) δ : 157.8(C-2), 105.9(C-3), 182.5(C-4), 155.3(C-5), 131.3(C-6), 163.6(C-7), 98.9(C-8), 148.9(C-9), 105.3(C-10), 126.9(C-1'), 129.3(C-2'), 126.2(C-3'), 132.0(C-4'), 126.2(C-5'), 129.3(C-6'), 62.1(C-OMe)。以上核磁数据与文献报道一致, 鉴定化合物**1**为金合欢素^[4]。

化合物**2**: 黄色无定形粉末, ESI-MS m/z 285 $[M+H]^+$, 提示分子式为 $C_{16}H_{12}O_5$; 1H -NMR(600 MHz, $CDCl_3$) δ : 6.72(1H, s, H-3), 6.48(1H, s, H-8), 7.96~7.92(2H, m, H-12, 16), 7.59(3H, dq, $J=8.7, 6.8$ Hz, H-13~15), 4.07(3H, s, OMe); ^{13}C -NMR(150 MHz, $CDCl_3$) δ : 163.6(C-2), 105.9(C-3), 182.5(C-4), 155.4(C-5), 98.9(C-6), 157.8(C-7), 126.2(C-8), 148.9(C-9), 105.3(C-10), 129.3(C-1'), 131.3(C-2'), 126.9(C-3'), 132.0(C-4'), 126.9(C-5'), 131.3(C-6'), 62.1(OMe)。以上核磁数据与文献报道一致, 鉴定化合物**2**为千层纸素A^[5]。

化合物**3**: 白色无定形粉末, ESI-MS m/z 296 $[M+H]^+$, 提示分子式为 $C_{18}H_{17}NO_3$; 1H -NMR(600 MHz, $CDCl_3$) δ : 7.52(1H, d, $J=15.4$ Hz, H-1), 6.15(1H, d, $J=15.4$ Hz, H-2), 3.69~3.60(2H, m, H-4), 2.88(2H, t, $J=6.8$ Hz, H-5), 6.96(2H, d, $J=8.7$ Hz, H-2'), 6.78(1H, d, $J=7.8$ Hz, H-5'), 6.96(2H, d, $J=8.7$ Hz, H-6'), 7.28~7.19(4H, m, H-2'', 3'', 5'', 6''), 7.32(1H, dd, $J=8.3, 6.9$ Hz, H-4''), 5.63(1H, d, $J=6.1$ Hz, NH), 5.97(2H, d, $J=1.3$ Hz, OCH_2O);

$^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ : 140.9(C-1), 118.5(C-2), 166.1(C-3), 40.8(C-4), 35.7(C-5), 126.6(C-1'), 109.7(C-2'), 149.1(C-3'), 150.6(C-4'), 111.1(C-5'), 122.0(C-6'), 139.0(C-1''), 127.8(C-2''), 128.8(C-3''), 126.6(C-4''), 128.7(C-5''), 127.8(C-6''), 56.0(OMe), 55.9(OMe)。以上核磁数据与文献报道一致, 鉴定化合物**3**为7E-3, 4-二氧甲基苯并[2-苯基-胸苷]^[6]。

化合物**4**: 白色无定形粉末, ESI-MS m/z 361 $[\text{M} + \text{H}]^+$, 提示分子式为 $\text{C}_{20}\text{H}_{24}\text{O}_6$; $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ : 2.57(1H, dd, $J = 13.7$, 10.7 Hz, H-7 α), 2.94(1H, dd, $J = 13.7$, 5.2 Hz, H-7 β), 2.81 ~ 2.70(1H, m, H-8), 3.78(2H, ddd, $J = 14.7$, 9.6, 6.3 Hz, H-9 α , 9' α), 4.07(1H, dd, $J = 8.6$, 6.6 Hz, H-9 β), 4.81(1H, d, $J = 6.6$ Hz, H-7'), 2.43(1H, p, $J = 6.9$ Hz, H-8'), 3.95 ~ 3.91(1H, m, H-9' β), 6.95 ~ 6.62(6H, m, H-Arom), 3.89(3H, s, 3-OMe), 3.91(3H, s, 3'-OMe); 132.3(C-1), 111.2(C-2), 146.5(C-3), 144.0(C-4), 114.4(C-5), 121.2(C-6), 134.8(C-1'), 108.3(C-2'), 146.6(C-3'), 145.1(C-4'), 114.2(C-5'), 118.8(C-6'), 33.4(C-7), 42.4(C-8), 72.9(C-9), 82.8(C-7'), 52.6(C-8'), 61.0(C-9'), 56.0(OMe)。以上核磁数据与文献报道一致, 鉴定化合物**4**为落叶松脂素^[7]。

化合物**5**: 白色无定形粉末, ESI-MS m/z 249 $[\text{M} + \text{H}]^+$, 提示分子式为 $\text{C}_{15}\text{H}_{20}\text{O}_3$; $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ : 1.73(1H, ddq, $J = 12.9$, 4.9, 2.3 Hz, H-1 α), 1.35(1H, dp, $J = 13.5$, 2.0 Hz, H-1 β), 1.99(1H, td, $J = 13.4$, 5.4 Hz, H-2 α), 1.68 ~ 1.57(1H, m, H-2 β), 2.45 ~ 2.39(1H, m, H-3 α), 2.39 ~ 2.32(2H, m, H-2 β , 6 β), 2.10(1H, td, $J = 13.6$, 4.5 Hz, H-5), 2.70(1H, dd, $J = 13.6$, 3.5 Hz, H-6 α), 3.92(1H, d, $J = 3.6$ Hz, H-8), 5.02(1H, dt, $J = 3.7$, 1.8 Hz, H-9), 3.51(3H, s, H-13), 0.93(3H, d, $J = 0.9$ Hz, H-14), 4.91(1H, q, $J = 1.5$ Hz, H-15 α), 4.63(1H, q, $J = 1.5$ Hz, H-15 β); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ : 41.0(C-1), 22.2(C-2), 36.2(C-3), 148.9(C-4), 42.2(C-5), 25.6(C-6), 159.1(C-7), 79.9(C-8), 75.4(C-9), 34.5(C-10), 121.9(C-11), 175.2(C-12), 8.3(C-13), 15.6(C-14), 107.4(C-15)。以上核磁数据与文献报道一致, 鉴定化合物**5**为9 α -羟基紫菀

内酯^[8]。

化合物**6**: 白色无定形粉末, ESI-MS m/z 733 $[\text{M} + \text{H}]^+$, 提示分子式为 $\text{C}_{40}\text{H}_{44}\text{O}_{13}$; $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ : 2.10 ~ 2.05(1H, m, H-1), 1.02(1H, overlapped, H-2 α), 0.35 ~ 0.30(1H, m, H-2 β), 1.85 ~ 1.82(2H, m, H-3), 3.98(1H, d, $J = 3.7$ Hz, H-6), 3.90(1H, s, H-9), 1.97(3H, s, H-13), 1.02(3H, s, H-14), 2.97 ~ 2.44(6H, m, H-15, 6', f, g), 1.63(1H, td, $J = 8.1$, 4.1 Hz, H-1'), 0.76(1H, td, $J = 8.8$, 5.8 Hz, H-2' α), 1.36(1H, dt, $J = 5.8$, 3.9 Hz, H-2' β), 1.42(1H, m, H-3'), 1.85 ~ 1.82(2H, m, H-5'), 1.90 ~ 1.88(1H, m, H-9'), 4.61 ~ 4.51(2H, m, H-13' α , 15' α), 5.12 ~ 5.05(2H, m, H-13' β , e), 0.84(3H, s, H-14'), 3.66(1H, d, $J = 11.8$ Hz, H-15' β), 6.64(1H, t, $J = 6.7$ Hz, H-c), 4.69 ~ 4.62(1H, m, H-d); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ : 26.0(C-1), 16.0(C-2), 24.8(C-3), 142.5(C-4), 132.2(C-5), 41.1(C-6), 131.3(C-7), 200.7(C-8), 79.9(C-9), 51.0(C-10), 147.5(C-11), 170.2(C-12), 20.1(C-13), 15.3(C-14), 25.3(C-15), 25.6(C-1'), 11.7(C-2'), 27.8(C-3'), 77.1(C-4'), 61.2(C-5'), 23.4(C-6'), 174.5(C-7'), 93.2(C-8'), 55.5(C-9'), 44.9(C-10'), 123.4(C-11'), 171.7(C-12'), 54.3(C-13'), 26.0(C-14'), 72.0(C-15'), 167.0(C-a), 129.2(C-b), 135.5(C-c), 61.6(C-d), 171.5(C-e), 28.6(C-f), 29.2(C-g), 172.0(C-h), 13.0(Me-b), 52.4(OMe)。以上核磁数据与文献报道一致, 鉴定化合物**6**为银线草醇B^[9]。

化合物**7**: 白色无定形粉末, ESI-MS m/z 767 $[\text{M} + \text{H}]^+$, 提示分子式为 $\text{C}_{40}\text{H}_{46}\text{O}_{15}$; $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ : 2.08 ~ 2.04(1H, m, H-1), 1.32 ~ 1.28(1H, m, H-2 α), 0.98(1H, td, $J = 8.4$, 5.8 Hz, H-2 β), 1.88 ~ 1.84(1H, m, H-3), 3.75(1H, overlapped, H-9), 1.83(3H, s, H-13), 1.06(3H, s, H-14), 3.08(1H, dd, $J = 14.2$, 7.1 Hz, H-15 α), 1.65 ~ 1.58(1H, m, H-15 β), 1.65 ~ 1.58(1H, m, H-1'), 1.26(1H, dt, $J = 5.7$, 4.1 Hz, H-2' α), 0.65(1H, td, $J = 9.0$, 5.6 Hz, H-2' β), 1.51 ~ 1.46(1H, m, H-3'), 1.65 ~ 1.58(1H, m, H-5'), 2.94 ~ 2.85(1H, m, H-6' α), 2.43 ~ 2.37(1H, m, H-6' β), 2.60 ~ 2.56(1H, m, H-9'),

5.27(1H, dd, $J = 12.2, 1.2$ Hz, H-13' α), 4.57(1H, d, $J = 12.2$ Hz, H-13' β), 0.94(3H, s, H-14'), 4.40(1H, d, $J = 11.8$ Hz, H-15' α), 3.89(1H, d, $J = 11.7$ Hz, H-15' β), 6.59(1H, ddt, $J = 6.4, 3.4, 1.0$ Hz, H-c), 5.16 ~ 5.08(1H, m, H-d α), 4.63(1H, ddd, $J = 14.4, 7.2, 1.1$ Hz, H-d β), 2.82 ~ 2.77(1H, m, H-f α), 2.67(1H, ddd, $J = 16.9, 8.1, 2.4$ Hz, H-f β), 2.94 ~ 2.85(1H, m, H-g α), 2.56 ~ 2.51(1H, m, H-g β), 1.95(3H, s, Me-b), 3.75(3H, s, OMe); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ : 26.0(C-1), 8.2(C-2), 27.7(C-3), 90.5(C-4), 158.5(C-5), 126.7(C-6), 142.4(C-7), 199.0(C-8), 77.8(C-9), 50.1(C-10), 129.5(C-11), 169.5(C-12), 20.7(C-13), 15.1(C-14), 36.8(C-15), 27.3(C-1'), 10.1(C-2'), 28.6(C-3'), 77.1(C-4'), 56.9(C-5'), 23.6(C-6'), 172.7(C-7'), 87.2(C-8'), 52.5(C-9'), 45.4(C-10'), 124.7(C-11'), 171.8(C-12'), 54.0(C-13'), 23.8(C-14'), 73.3(C-15'), 166.7(C-a), 129.5(C-b), 135.6(C-c), 61.4(C-d), 171.5(C-e), 29.1(C-f), 28.7(C-g), 172.0(C-h), 12.8(Me-b), 52.5(OMe)。以上核磁数据与文献报道一致, 鉴定化合物**7**为金粟兰素C^[10]。

化合物**8**: 白色无定形粉末, ESI-MS m/z 249 $[\text{M} + \text{H}]^+$, 提示分子式为 $\text{C}_{15}\text{H}_{20}\text{O}_3$, $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ : 1.25(1H, td, $J = 12.7, 5.7$ Hz, H-1 α), 1.70 ~ 1.53(3H, m, H-1 β , 6 β , 9 α), 1.97(1H, td, $J = 12.7, 7.0$ Hz, H-2 β), 2.37(1H, ddt, $J = 15.1, 4.2, 1.8$ Hz, H-3 α), 2.43(1H, tq, $J = 13.0, 1.5$ Hz, H-3 β), 2.62(1H, dd, $J = 13.2, 3.2$, H-6 α), 2.26(1H, d, $J = 13.7$ Hz, H-9 β), 1.89 ~ 1.80(4H, m, H-5, 13), 1.03(3H, s, H-14), 4.60(1H, q, $J = 1.5$ Hz, H-15 α), 4.87(1H, q, $J = 1.5$ Hz, H-15 β); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ : 51.3(C-1), 22.3(C-2), 36.7(C-3), 148.5(C-4), 51.3(C-5), 24.6(C-6), 160.8(C-7), 103.4(C-8), 41.3(C-9), 36.1(C-10), 122.3(C-11), 171.9(C-12), 8.2(C-13), 16.6(C-14), 106.9(C-15)。以上核磁数据与文献报道一致, 鉴定化合物**8**为白术内酯III^[11]。

化合物**9**: 白色无定形粉末, ESI-MS m/z 233 $[\text{M} + \text{H}]^+$, 提示分子式为 $\text{C}_{15}\text{H}_{20}\text{O}_2$, $^1\text{H-NMR}$

(600 MHz, CDCl_3) δ : 1.34(1H, td, $J = 13.3, 12.9, 5.2$ Hz, H-1 α), 1.72 ~ 1.56(3H, m, H-1 β , 2), 2.04 ~ 1.96(1H, m, H-3 α), 2.40(1H, ddt, $J = 13.2, 4.2, 2.0$ Hz, H-3 β), 1.89 ~ 1.85(1H, m, H-5), 2.75(1H, dd, $J = 13.9, 3.7$ Hz, H-6 α), 2.36 ~ 2.29(2H, m, H-6 β , 9 β), 4.87 ~ 4.81(1H, m, H-8), 1.19 ~ 1.12(1H, m, H-9 α), 1.84(3H, t, $J = 1.7$ Hz, H-13), 0.92(3H, s, H-14), 4.62(1H, q, $J = 1.5$ Hz, H-15 α), 4.89(1H, q, $J = 1.6$ Hz, H-15 β); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ : 41.1(C-1), 22.4(C-2), 36.3(C-3), 149.3(C-4), 41.5(C-5), 25.7(C-6), 162.0(C-7), 78.0(C-8), 47.6(C-9), 37.0(C-10), 119.5(C-11), 174.2(C-12), 8.3(C-13), 16.5(C-14), 106.9(C-15)。以上核磁数据与文献报道一致, 鉴定化合物**9**为白术内酯II^[12]。

4 讨论

本研究在银线草75%乙醇提取物的乙酸乙酯溶性部位中分离得到9个化合物, 其中化合物**1**~**4**为首次在金粟兰属中分离得到, 丰富了金粟兰属的化学成分, 为其药理活性研究提供参考。

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