倒挂金钩茎枝的化学成分研究

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摘 要：目的 研究倒挂金钩 *Uncaria lancifolia* 茎枝的化学成分。**方法** 采用硅胶、RP_{18}、Sephadex LH-20 等柱色谱和高效液相色谱技术进行化合物的分离和纯化，根据理化性质和波谱数据鉴定化合物的结构。**结果** 从倒挂金钩茎枝的 95%乙醇提取物中分离得到 18 个化合物，分别鉴定为台钩藤碱 A（**1**）、台钩藤碱 E（**2**）、异帽柱木菲碱（**3**）、四氢脚鸭木碱（**4**）、异胡豆苷（**5**）、卡丹宾碱（**6**）、glabratine（**7**）、异长春花苷内酰胺（**8**）、(13*R*)-hydroxy-octodeca-(9*Z*,11*E*,15*Z*)-trien-oic acid（**9**）、(6*S*,9*R*)-长寿花糖苷（**10**）、苯甲酰基-β-*D*-吡喃葡萄糖苷（**11**）、integracin A（**12**）、integracin B（**13**）、6β,19α-二羟基乌苏-3-氧代-12-烯-28-酸（**14**）、乌苏酸（**15**）、齐墩果酸（**16**）、β-谷甾醇（**17**）、β-胡萝卜苷（**18**）。**结论** 首次对倒挂金钩中的化学成分进行了研究，所有化合物均首次从该植物中分离得到，化合物 **9**～**13** 首次从该属植物中分离得出。化合物 **1**～**8** 为单萜吲哚生物碱，是钩藤属植物中的特征性成分。

关键词：倒挂金钩；单萜吲哚生物碱；台钩藤碱 A；台钩藤碱 E；异帽柱木菲碱；(6*S*,9*R*)-长寿花糖苷

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Chemical constituents from stems of *Uncaria lancifolia*

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Abstract: Objective To study the chemical constituents from the stems of *Uncaria lancifolia*. **Methods** The isolation and purification were carried out by silica gel column chromatography, RP_{18}, Sephadex LH-20, and preparative HPLC. The structures of the isolated compounds were elucidated by physical and chemical properties, and spectroscopic methods. **Results** Eighteen compounds were isolated and identified from 95% ethanol extract from the stems of *U. lancifolia* and characterized as uncarine A (**1**), uncarine E (**2**), isomitraphylline (**3**), tetrahydroalstonine (**4**), strictosidine (**5**), cadambine (**6**), glabratine (**7**), strictosamide (**8**), (13*R*)-hydroxy-octodeca-(9*Z*,11*E*,15*Z*)-trien-oic acid (**9**), (6*S*,9*R*)-roseoside (**10**), periplanetin (**11**), integracin A (**12**), integracin B (**13**), 6β,19α-dihydroxyurs-3-oxours-12-en-28-oic aicd (**14**), ursolic acid (**15**), oleanic acid (**16**), β-sitosterol (**17**), and β-daucosterol (**18**). **Conclusion** This is the first report for the chemical constituents from *U. lancifolia*. All compounds are obtained from this plant for the first time, and compounds **9**—**13** are isolated from *Uncaria* genus for the first time. Compounds **1**—**8** are monoterpene indole alkaloids, which are characteristic constituents in *Uncaria* genus.

Key words: *Uncaria lancifolia* Hutchins.; monoterpene indole alkaloids; uncarine A; uncarine E; isomitraphylline; (6*S*,9*R*)-roseoside

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倒挂金钩 *Uncaria lancifolia* Hutchins.，又名披针叶钩藤，为茜草科（Rubiaceae）钩藤属 *Uncaria* Schreber nom. cons. 植物，其节、钩、茎用于清热、平肝、镇惊，带钩枝条可治高血压、头晕、目眩、妇人子痫、乳腺炎等，在云南部分地区作中药钩藤使用[1]。关于倒挂金钩化学成分的研究，迄今未见相关报道。本实验对倒挂金钩茎枝的化学成分进行研究，从 95%乙醇提取物中分离得到 18 个化合物，包括 8 个单萜吲哚生物碱和 10 个其他类化合物，分别鉴定为台钩藤碱 A（uncarine A，**1**）、台钩藤碱 E（uncarine E，**2**）、异帽柱木菲碱（isomitraphylline，**3**）、四氢脚鸭木碱（tetrahydroalstonine，**4**）、异胡豆苷（strictosidine，**5**）、卡丹宾碱（cadambine，**6**）、glabratine（**7**）、异长春花苷内酰胺（strictosamide，**8**）、(13*R*)-hydroxy-octodeca-(9*Z*,11*E*,15*Z*)-trien-oic acid（**9**）、(6*S*,9*R*)-长寿花糖苷 [(6*S*,9*R*)-roseoside，**10**]、苯甲酰基-β-*D*-吡喃葡萄糖苷（periplanetin，**11**）、integracin A（**12**）、integracin B（**13**）、6β,19α-二羟基乌苏-3-氧代-12-烯-28-酸（6β,19α-dihydroxyurs-3-oxours-12-en-28-oic aicd，**14**）、乌苏酸（ursolic acid，**15**）、齐墩果酸（oleanic acid，**16**）、β-谷甾醇（β-sitosterol，**17**）、β-胡萝卜苷（β-daucosterol，**18**）。所有化合物均首次从该植物中分离得到，其中化合物 **9**～**13** 首次从该属植物中分离得到。

1 仪器与材料

UPLC-IT-TOF 液相-离子阱飞行时间色谱质谱联用仪（日本岛津制作所）；Agilent G6230 飞行时间质谱仪、Agilent 1200 分析型和制备型高效液相色谱仪（美国 Agilent 公司）；AVANCEIII 500 MHz 核磁共振仪（德国 Brucker 公司）；BUCHI pump Manager C-615 中压反相色谱仪（瑞士 Buchi 公司）；WFH-203（2F-1）三用紫外分析仪（上海精科实业有限公司）；Lichroprep RP_{18} gel、Sephadex LH-20 葡聚糖凝胶（德国 Merck 公司）；柱色谱硅胶与薄层色谱硅胶板 GF_{254}（青岛海洋化工厂）；所有试剂均为色谱纯或分析纯；对照品乌苏酸（批号 bbp00038）、齐墩果酸（批号 bbp00607）、β-谷甾醇（批号 bbp03116）和 β-胡萝卜苷（批号 bbp00055）均为云南西力生物公司产品，质量分数均大于 98%。

倒挂金钩茎枝采于云南省文山州马关县，原植物经中国医学科学院药用植物研究所云南分所西双版纳州傣药南药重点实验室李海涛副研究员鉴定为茜草科钩藤属植物倒挂金钩 *Uncaria lancifolia* Hutchins.。标本（U-2016-001）保存于昆明医科大学药学院药物化学教研室。

2 提取与分离

20 kg 倒挂金钩茎枝，粉碎后用 95%工业乙醇室温浸泡提取 3 次，每次 7 d，减压浓缩得总浸膏 1.5 kg。用硅胶（80～100 目）拌样，经硅胶柱色谱，用石油醚-丙酮（10∶1→0∶1）系统洗脱，TLC 跟踪，合并相似部分得到 5 个组分 Fr. A～E。Fr. A（3.3 g）析出晶体，重结晶纯化得到化合物 **17**（465.2 mg）；Fr. B（54.3 g）经硅胶柱色谱，用氯仿-甲醇（10∶1→0∶1）梯度洗脱得到 3 个亚流分 Fr. B1～B3。Fr. B1 经硅胶、凝胶柱色谱（甲醇）纯化得到化合物 **14**（69.2 mg）。Fr. B2 经硅胶柱色谱（氯仿-甲醇 100∶1→60∶1）得到化合物 **2**（50.2 mg）和 **4**（76.4 mg），再经硅胶柱色谱，以氯仿-甲醇（100∶1→10∶1）梯度洗脱，用 Sephadex LH-20 凝胶（甲醇）、制备型 HPLC（甲醇-水 40∶60）纯化得到化合物 **1**（214.8 mg）和 **3**（198.2 mg），再经 RP_{18} 柱色谱常压分离，用甲醇水（50%～70%）梯度洗脱，反复硅胶、凝胶柱色谱得到化合物 **9**（40.5 mg）、**15**（52.3 mg）和 **16**（30.3 mg）。Fr. B3 经重结晶得化合物 **18**（1.2 g）。Fr. C（16.4 g）经硅胶柱色谱，氯仿-甲醇（60∶1→10∶1）梯度洗脱，再经制备型 HPLC（甲醇-水 25∶75）制备得到化合物 **11**（20.2 mg）。Fr. D（250.8 g）经硅胶柱色谱，用氯仿-甲醇（10∶1→0∶1）梯度洗脱得到 3 个亚流分 Fr. D1～D3。Fr. D1 经 Sephadex LH-20 凝胶柱色谱（甲醇）纯化得到化合物 **10**（10.6 mg）。Fr. D2 用制备型 HPLC（甲醇-水 20∶80）制备得到化合物 **7**（56.5 mg）。Fr. D3 经反复硅胶、Sephadex LH-20 凝胶柱色谱（甲醇）纯化得到化合物 **12**（29.7 mg）和 **13**（25.4 mg）。Fr. E（300g）经硅胶柱色谱，用氯仿-甲醇（5∶1→0∶1）梯度洗脱分离得到 3 个亚流分 Fr. E1～E3。Fr. E1 经硅胶柱色谱、Sephadex LH-20 凝胶柱色谱（甲醇-氯仿 1∶1）和制备型 HPLC（甲醇-水 55∶45）纯化得到化合物 **6**（45.6 mg）。Fr. E2 经反复硅胶柱色谱和制备型 HPLC（甲醇-水 50∶50）纯化得到化合物 **5**（98.7 mg）和 **8**（15.1 mg）。

3 结构鉴定

化合物 **1**：白色无定形粉末，ESI-MS *m*/*z*: 369 $[M+H]^+$。^{1}H-NMR (500 MHz, $CDCl_3$) δ: 2.60 (1H, dd, J = 11.5, 2.2 Hz, H-3), 2.57 (1H, t, J = 8.6 Hz,

H-5α), 3.31 (1H, td, *J* = 8.6, 2.4 Hz, H-5β), 2.05 (1H, m, H-6α), 2.40 (1H, ddd, *J* = 13.0, 8.6, 2.4 Hz, H-6β), 7.35 (1H, d, *J* = 7.3 Hz, H-9), 7.00 (1H, td, *J* = 7.6, 0.8 Hz, H-10), 7.18 (1H, td, *J* = 7.6, 1.3 Hz, H-11), 6.86 (1H, d, *J* = 7.8 Hz, H-12), 2.16 (1H, m, H-14α), 0.56 (1H, t, *J* = 11.5 Hz, H-14β), 2.16 (1H, m, H-15), 7.41 (1H, d, *J* = 1.4 Hz, H-17), 1.33 (3H, d, *J* = 6.4 Hz, 18-CH_3), 3.81 (1H, m, H-19), 1.51 (1H, m, H-20), 1.91 (1H, t, *J* = 10.8 Hz, H-21α), 3.26 (1H, dd, *J* = 10.8, 3.3 Hz, H-21β), 3.56 (3H, s, 23-OCH_3), 8.12 (1H, brs, NH)；^{13}C-NMR (125 MHz, $CDCl_3$) *δ*: 181.3 (C-2), 71.2 (C-3), 53.4 (C-5), 35.4 (C-6), 56.4 (C-7), 133.9 (C-8), 124.9 (C-9), 122.4 (C-10), 127.6 (C-11), 109.6 (C-12), 140.2 (C-13), 29.2 (C-14), 36.0 (C-15), 108.7 (C-16), 155.3 (C-17), 18.4 (18-CH_3), 75.6 (C-19), 43.6 (C-20), 53.4 (C-21), 167.1 (C-22), 50.8 (23-OCH_3)。以上数据与文献对照[2]，鉴定化合物 **1** 为台钩藤碱 A。

化合物 **2**：淡黄色油状物，ESI-MS *m*/*z*: 369 [M＋H]$^+$。^{1}H-NMR (500 MHz, $CDCl_3$) *δ*: 2.57 (1H, dd, *J* = 11.7, 2.8 Hz, H-3), 2.49 (1H, m, H-5α), 3.21 (1H, td, *J* = 7.6, 2.4 Hz, H-5β), 1.99 (1H, m, H-6α), 2.39 (1H, dd, *J* = 11.9, 7.6 Hz, H-6β), 7.27 (1H, d, *J* = 7.7 Hz, H-9), 7.03 (1H, td, *J* = 7.7, 1.1 Hz, H-10), 7.19 (1H, td, *J* = 7.7, 1.3 Hz, H-11), 6.87 (1H, d, *J* = 7.7 Hz, H-12), 1.61 (1H, m, H-14α), 0.87 (1H, t, *J* = 11.7 Hz, H-14β), 2.50 (1H, ddd, *J* = 11.7, 2.8, 2.8 Hz, H-15), 7.40 (1H, s, H-17), 1.33 (3H, d, *J* = 6.2 Hz, 18-CH_3), 4.35 (1H, m, H-19), 1.59 (1H, m, H-20), 2.40 (1H, dd, *J* = 11.9, 3.8 Hz, H-21α), 3.29 (1H, dd, *J* = 11.9, 1.8 Hz, H-21β), 3.60 (3H, s, 23-OCH_3), 8.40 (1H, brs, NH)；^{13}C-NMR (125 MHz, $CDCl_3$) *δ*: 181.3 (C-2), 71.3 (C-3), 54.2 (C-5), 34.9 (C-6), 56.9 (C-7), 133.8 (C-8), 124.6 (C-9), 122.6 (C-10), 127.7 (C-11), 109.6 (C-12), 140.2 (C-13), 30.2 (C-14), 30.5 (C-15), 109.8 (C-16), 155.0 (C-17), 18.7 (18-CH_3), 72.2 (C-19), 37.9 (C-20), 53.5 (C-21), 167.7 (C-22), 51.0 (23-OCH_3)。以上数据与文献对照[2]，鉴定化合物 **2** 为台钩藤碱 E。

化合物 **3**：白色无定形粉末，ESI-MS *m*/*z*: 369 [M＋H]$^+$。^{1}H-NMR (500 MHz, $CDCl_3$) *δ*: 2.60 (1H, dd, *J* = 11.5, 3.0 Hz, H-3), 2.53 (1H, t, *J* = 8.9 Hz, H-5α), 3.30 (1H, td, *J* = 8.9, 2.3 Hz, H-5β), 2.04 (1H, m, H-6α), 2.40 (1H, ddd, *J* = 13.0, 8.9, 2.3 Hz, H-6β), 7.35 (1H, d, *J* = 7.6 Hz, H-9), 7.00 (1H, td, *J* = 7.6, 0.8 Hz, H-10), 7.17 (1H, td, *J* = 7.6, 1.2 Hz, H-11), 6.85 (1H, d, *J* = 7.6 Hz, H-12), 2.22 (1H, m, H-14α), 0.59 (1H, t, *J* = 11.5 Hz, H-14β), 2.18 (1H, m, H-15), 7.38 (1H, d, *J* = 1.7 Hz, H-17), 1.12 (3H, d, *J* = 6.9 Hz, CH_3-18), 4.37 (1H, m, H-19), 1.93 (1H, m, H-20), 1.94 (1H, dd, *J* = 11.0, 11.0 Hz, H-21α), 3.12 (1H, dd, *J* = 11.0, 7.30 Hz, H-21β), 3.58 (3H, s, 23-OCH_3), 7.81 (1H, brs, NH)；^{13}C-NMR (125 MHz, $CDCl_3$) *δ*: 181.0 (C-2), 71.8 (C-3), 53.4 (C-5), 35.4 (C-6), 56.3 (C-7), 133.8 (C-8), 124.9 (C-9), 122.4 (C-10), 127.6 (C-11), 109.5 (C-12), 140.1 (C-13), 14.9 (C-14), 30.0 (C-15), 107.4 (C-16), 153.9 (C-17), 14.9 (18-CH_3), 74.0 (C-19), 40.9 (C-20), 54.3 (C-21), 167.1 (C-22), 50.8 (23-OCH_3)。以上数据与文献对照[2]，鉴定化合物 **3** 为异帽柱木菲碱。

化合物 **4**：黄色无定形粉末，ESI-MS *m*/*z*: 353 [M＋H]$^+$。^{1}H-NMR (500 MHz, $CDCl_3$) *δ*: 3.34 (1H, d, *J* = 10.9 Hz, H-3), 2.78 (1H, m, H-5α), 2.96 (1H, m, H-5β), 2.68 (1H, m, H-6α), 2.71 (1H, m, H-6β), 7.45 (1H, d, *J* = 7.7 Hz, H-9), 7.09 (1H, td, *J* = 7.7, 1.1 Hz, H-10), 7.10 (1H, td, *J* = 7.7, 1.3 Hz, H-11), 7.26 (1H, d, *J* = 7.7 Hz, H-12), 1.70 (1H, m, H-14α), 1.26 (1H, m, H-14β), 2.53 (1H, m, H-15), 7.57 (1H, s, H-17), 1.41 (3H, d, *J* = 6.2 Hz, 18-CH_3), 4.50 (1H, m, H-19), 1.45 (1H, m, H-20), 2.50 (1H, m, H-21α), 2.55 (1H, m, H-21β), 3.76 (3H, s, 23-OCH_3), 7.92 (1H, brs, NH)；^{13}C-NMR (125 MHz, $CDCl_3$) *δ*: 134.5 (C-2), 38.4 (C-3), 53.5 (C-5), 34.2 (C-6), 109.5 (C-7), 127.1 (C-8), 121.3 (C-9), 110.8 (C-10), 119.3 (C-11), 118.0 (C-12), 136.0 (C-13), 21.7 (C-14), 31.3 (C-15), 108.0 (C-16), 155.8 (C-17), 18.5 (18-CH_3), 72.4 (C-19), 59.8 (C-20), 56.2 (C-21), 168.0 (C-22), 51.1 (23-OCH_3)。以上数据与文献对照[3]，鉴定化合物 **4** 为四氢脚鸭木碱。

化合物 **5**：黄色无定形粉末，ESI-MS *m*/*z*: 531 [M＋H]$^+$。^{1}H-NMR (500 MHz, CD_3OD) *δ*: 4.40 (1H, dd, *J* = 11.4, 3.0 Hz, H-3), 3.20 (1H, m, H-5α), 3.50 (1H, m, H-5β), 2.93 (1H, m, H-6α), 3.05 (1H, m, H-6β), 7.41 (1H, dd, *J* = 7.9, 1.2 Hz, H-9), 6.98 (1H, td, *J* = 7.1, 1.1 Hz, H-10), 7.07 (1H, td, *J* = 7.1, 1.2 Hz, H-11), 7.28 (1H, dd, *J* = 8.1, 1.1 Hz, H-12), 2.24 (1H, ddd, *J* = 14.7, 11.4, 3.9 Hz, H-14α), 2.14 (1H, ddd, *J* = 14.7, 11.5, 3.0 Hz, H-14β), 3.05 (1H, m,

H-15), 7.75 (1H, s, H-17), 5.33 (1H, dd, *J* = 17.9, 2.1 Hz, H-18*Z*), 5.24 (1H, dd, *J* = 10.6, 2.1 Hz, H-18*E*), 5.84 (1H, m, H-19), 2.72 (1H, m, H-20), 5.85 (1H, m, H-21), 3.77 (3H, s, 23-OCH_3), 4.83 (1H, d, *J* = 7.9 Hz, H-1′), 3.31～3.56 (4H, m, H-2′～5′), 3.97 (1H, dd, *J* = 11.9, 2.1 Hz, H-6′α), 3.64 (1H, dd, *J* = 11.9, 6.7 Hz, H-6′β)；^{13}C-NMR (125 MHz, CD_3OD) *δ*: 133.2 (C-2), 52.4 (C-3), 42.9 (C-5), 21.0 (C-6), 107.7 (C-7), 127.9 (C-8), 118.9 (C-9), 120.1 (C-10), 122.7 (C-11), 112.0 (C-12), 137.9 (C-13), 35.9 (C-14), 32.4 (C-15), 109.9 (C-16), 156.1 (C-17), 119.5 (C-18), 135.7 (C-19), 45.6 (C-20), 97.5 (C-21), 170.6 (C-22), 52.4 (23-OCH_3), 100.3 (C-1′), 78.6 (C-2′), 78.0 (C-3′), 74.6 (C-4′), 71.7 (C-5′), 62.9 (C-6′)。以上数据与文献对照[4-5]，又通过 ^{1}H-^{1}H ROESY、^{1}H-^{13}C HMBC 和 ^{1}H-^{13}C HSQC 确定立体构型，鉴定化合物 **5** 为异胡豆苷。

化合物 **6**：黄色无定形粉末，ESI-MS *m*/*z*: 545 $[M+H]^+$。^{1}H-NMR (500 MHz, CD_3OD) *δ*: 2.79 (1H, m, H-5α), 3.16 (1H, m, H-5β), 2.79 (2H, m, H-6), 7.42 (1H, dd, *J* = 7.9, 1.2 Hz, H-9), 6.99 (1H, td, *J* = 7.1, 1.1 Hz, H-10), 7.09 (1H, td, *J* = 7.1, 1.2 Hz, H-11), 7.29 (1H, dd, *J* = 8.1, 1.1 Hz, H-12), 2.42 (1H, m, H-14α), 2.14 (1H, m, H-14β), 3.00 (1H, m, H-15), 7.75 (1H, s, H-17), 3.03 (1H, m, H-18α), 3.52 (1H, d, *J* = 10.8 Hz, H-18β), 4.93 (1H, m, H-19), 1.76 (1H, m, H-20), 5.83 (1H, d, *J* = 9.3 Hz, H-21), 3.64 (3H, s, OCH_3-23), 4.79 (1H, d, *J* = 11.0 Hz, H-1′), 3.32～3.43 (4H, m, H-2′～5′), 3.62 (1H, m, H-6′α), 3.85 (1H, d, *J* = 2.1 Hz, H-6′β)；^{13}C-NMR (125 MHz, CD_3OD) *δ*: 133.0 (C-2), 92.9 (C-3), 53.7 (C-5), 22.6 (C-6), 111.5 (C-7), 126.8 (C-8), 120.1 (C-9), 119.7 (C-10), 123.3 (C-11), 112.4 (C-12), 138.4 (C-13), 42.9 (C-14), 26.7 (C-15), 111.1 (C-16), 154.2 (C-17), 97.5 (C-18), 40.9 (C-19), 74.7 (C-20), 59.3 (C-21), 168.8 (C-22), 51.7 (23-OCH_3), 101.5 (C-1′), 74.7 (C-2′), 78.4 (C-3′), 71.6 (C-4′), 78.4 (C-5′), 62.8 (C-6′)。以上数据与文献对照[6]，鉴定化合物 **6** 为卡丹宾碱。

化合物 **7**：黄色油状物，ESI-MS *m*/*z*: 553 $[M+Na]^+$。^{1}H-NMR (500 MHz, DMSO-d_6) *δ*: 4.53 (1H, d, *J* = 13.6 Hz, H-3), 3.78 (1H, d, *J* = 12.0 Hz, H-5α), 3.43 (1H, m, H-5β), 3.95 (1H, brs, H-6), 6.61 (1H, d, *J* = 8.4 Hz, H-10), 6.89 (1H, t, *J* = 8.4 Hz, H-11), 6.94 (1H, d, *J* = 8.4 Hz, H-12), 2.60 (1H, d, *J* = 13.6 Hz, H-14α), 1.33 (1H, td, *J* = 13.6, 4.8 Hz, H-14β), 2.83 (1H, m, H-15), 7.57 (1H, s, H-17), 4.99 (1H, dd, *J* = 9.8, 2.0 Hz, H-18*Z*), 4.93 (1H, d, *J* = 9.8 Hz, H-18*E*), 5.74 (1H, m, H-19), 2.23 (1H, m, H-20), 3.54 (1H, m, H-21), 3.53 (3H, s, 23-OCH_3), 4.91 (1H, d, *J* = 7.2 Hz, H-1′), 3.26～3.30 (4H, m, H-2′～5′), 3.17 (1H, m, H-4′), 3.67 (1H, dd, *J* = 11.4, 4.2 Hz, H-6′α), 3.47 (1H, m, H-6′β)；^{13}C-NMR (125 MHz, DMSO-d_6) *δ*: 132.3 (C-2), 48.1 (C-3), 49.9 (C-5), 23.6 (C-6), 106.2 (C-7), 117.2 (C-8), 151.7 (C-9), 103.0 (C-10), 121.5 (C-11), 105.4 (C-12), 137.7 (C-13), 31.5 (C-14), 30.7 (C-15), 94.4 (C-16), 146.2 (C-17), 115.2 (C-18), 140.5 (C-19), 52.7 (C-20), 62.8 (C-21), 168.0 (C-22), 50.2 (23-OCH_3), 100.8 (C-1′), 73.6 (C-2′), 76.9 (C-3′), 69.8 (C-4′), 77.0 (C-5′), 60.7 (C-6′)。以上数据与文献对照[7]，鉴定化合物 **7** 为 glabratine。

化合物 **8**：黄色无定形粉末，ESI-MS *m*/*z*: 499 $[M+H]^+$。^{1}H-NMR (500 MHz, CD_3OD) *δ*: 5.02 (1H, m, H-3), 3.06 (1H, m, H-5α), 4.92 (1H, dd, *J* = 13.0, 5.5 Hz, H-5β), 2.66 (1H, m, H-6α), 2.96 (1H, m, H-6β), 7.32 (1H, dd, *J* = 7.7, 1.2 Hz, H-9), 6.98 (1H, td, *J* = 7.7, 1.1 Hz, H-10), 7.07 (1H, td, *J* = 7.7, 1.3 Hz, H-11), 7.32 (1H, d, *J* = 7.7 Hz, H-12), 2.02 (1H, m, H-14α), 2.44 (1H, d, *J* = 13.8 Hz, H-14β), 2.77 (1H, m, H-15), 7.37 (1H, d, *J* = 2.4 Hz, H-17), 5.31 (1H, dd, *J* = 10.0, 1.8 Hz, H-18*E*), 5.39 (3H, dd, *J* = 17.3, 1.8 Hz, H-18*Z*), 5.63 (1H, m, H-19), 2.67 (1H, m, H-20), 5.38 (1H, d, *J* = 1.6 Hz, H-21α), 4.57 (1H, d, *J* = 7.9 Hz, H-1′), 2.96 (1H, m, H-2′), 3.16～3.30 (3H, m, H-3′～5′), 3.62 (1H, dd, *J* = 11.8, 5.5 Hz, H-6′α), 3.85 (1H, dd, *J* = 11.8, 1.6 Hz, H-6′β)；^{13}C-NMR (125 MHz, CD_3OD) *δ*: 134.7 (C-2), 55.0 (C-3), 44.6 (C-5), 22.1 (C-6), 110.4 (C-7), 128.6 (C-8), 118.7 (C-9), 120.1 (C-10), 122.4 (C-11), 112.3 (C-12), 137.6 (C-13), 27.2 (C-14), 24.8 (C-15), 109.1 (C-16), 149.1 (C-17), 120.6 (C-18), 134.3 (C-19), 44.7 (C-20), 98.0 (C-21), 169.0 (C-22), 100.4 (C-1′), 74.3 (C-2′), 78.2 (C-3′), 71.4 (C-4′), 78.0 (C-5′), 62.6 (C-6′)。以上数据与文献对照[8]，鉴定化合物 **8** 为异长春花苷内酰胺。

化合物 **9**：黄色油状物，ESI-MS *m*/*z*: 317 $[M+Na]^+$。^{1}H-NMR (500 MHz, $CDCl_3$) *δ*: 2.35 (2H, m, H-2), 1.62 (1H, m, H-3), 1.32 (6H, m, H-4～6), 1.37

(2H, m H-7), 2.17 (2H, m, H-8), 5.44 (1H, m, H-9), 5.96 (1H, td, *J* = 10.7, 15.1 Hz, H-10), 6.52 (1H, dd, *J* = 15.4, 10.7 Hz, H-11), 5.68 (1H, dd, *J* = 15.4, 6.4 Hz, H-12), 4.21 (1H, dd, *J* = 6.4, 5.9 Hz, H-13), 2.35 (2H, m, H-14), 5.36 (3H, m, H-15), 5.55 (3H, m, H-16), 2.07 (2H, m, H-17), 0.97 (3H, t, *J* = 7.6 Hz, H-18)；^{13}C-NMR (125 MHz, $CDCl_3$) *δ*: 179.6 (C-1), 34.0 (C-2), 24.6 (C-3), 28.8 (C-4), 28.8 (C-5), 28.8 (C-6), 29.4 (C-7), 27.6 (C-8), 132.9 (C-9), 127.8 (C-10), 125.9 (C-11), 134.9 (C-12), 72.2 (C-13), 35.2 (C-14), 123.7 (C-15), 135.1 (C-16), 20.8 (C-17), 14.2 (C-18)。以上数据与文献对照[9]，鉴定化合物 **9** 为 (13*R*)-hydroxy-octodeca-(9*Z*,11*E*,15*Z*)-trien-oic acid。

化合物 **10**：棕黄色油状物，ESI-MS *m*/*z*: 409 $[M+Na]^+$。^{1}H-NMR (500 MHz, CD_3OD) *δ*: 2.16 (1H, dd, *J* = 17.0, 1.0 Hz, H-2α), 2.49 (1H, d, *J* = 17.0 Hz, H-2β), 5.86 (1H, m, H-4), 5.85 (1H, brs, H-7), 5.85 (2H, brs, H-8), 4.42 (1H, m, H-9), 1.28 (3H, d, *J* = 6.5 Hz, H-10), 1.00 (3H, s, H-11), 1.02 (3H, s, H-12), 1.91 (3H, d, *J* = 1.4 Hz, H-13), 4.34 (1H, d, *J* = 7.8 Hz, H-1′), 3.00～3.45 (4H, m, H-2′～5′), 3.63 (1H, dd, *J* = 12.0, 6.0 Hz, H-6′α), 3.85 (1H, dd, *J* = 12.0, 2.5 Hz, H-6′β)；^{13}C-NMR (125 MHz, CD_3OD) *δ*: 42.5 (C-1), 50.7 (C-2), 201.3 (C-3), 127.2 (C-4), 167.3 (C-5), 80.0 (C-6), 131.5 (C-7), 135.3 (C-8), 77.4 (C-9), 21.3 (C-10), 23.5 (C-11), 24.8 (C-12), 19.6 (C-13), 102.7 (C-1′), 75.4 (C-2′), 78.0 (C-3′), 71.6 (C-4′), 77.9 (C-5′), 62.8 (C-6′)。以上数据与文献对照[10]，鉴定化合物 **10** 为 (6*S*,9*R*)-长寿花糖苷。

化合物 **11**：黄色油状物，ESI-MS *m*/*z*: 307 $[M+Na]^+$。1H-NMR (500 MHz, CD_3OD) *δ*: 8.08 (2H, dd, *J* = 8.4, 1.3 Hz, H-2, 6), 7.49 (2H, t, *J* = 8.0 Hz, H-3, 5), 7.62 (1H, m, H-4), 5.74 (1H, d, *J* = 7.8 Hz, H-1′), 3.40～3.52 (4H, m, H-2～5′), 3.72 (1H, dd, *J* = 12.0, 6.0 Hz, H-6′α), 3.87 (1H, dd, *J* = 12.0, 2.5 Hz, H-6′β)；^{13}C-NMR (125 MHz, CD_3OD) *δ*: 130.8 (C-1), 130.9 (C-2, 6), 129.6 (C-3, 5), 134.8 (C-4), 166.8 (C-7), 96.3 (C-1′), 74.0 (C-2′), 78.0 (C-3′), 71.0 (C-4′), 78.9 (C-5′), 62.3 (C-6′)。以上数据与文献对照[11]，鉴定化合物 **11** 为苯甲酰基-β-*D*-吡喃葡萄糖苷。

化合物 **12**：黄色油状物，ESI-MS *m*/*z*: 629 $[M+H]^+$。^{1}H-NMR (500 MHz, $CDCl_3$) *δ*: 6.21 (1H, brs, H-1), 6.21 (1H, brs, H-3), 6.21 (1H, brs, H-5), 2.40 (1H, t, *J* = 7.4 Hz, H-7α), 1.50 (1H, m, H-8β), 1.26 (4H, m, H-9～12), 1.63 (1H, m, H-13), 5.23 (1H, m, H-14), 1.63 (1H, m, H-15), 1.40 (1H, m, H-16), 0.89 (1H, t, *J* = 7.3 Hz, H-17), 6.31 (1H, d, *J* = 2.5 Hz, H-4′α), 6.23 (1H, d, *J* = 2.5 Hz, H-6′α), 2.80 (1H, m, H-8′β), 1.50 (1H, m, H-9′β), 1.26 (4H, m, H-10′～13′), 1.50 (1H, m, H-14′), 4.91 (1H, m, H-15′), 1.50 (1H, m, H-16′), 1.37 (1H, m, H-17), 0.92 (1H, t, *J* = 7.3 Hz, H-18′α)；^{13}C-NMR (125 MHz, $CDCl_3$) *δ*: 107.9 (C-1), 156.7 (C-2), 100.3 (C-3), 156.7 (C-4), 107.9 (C-5), 145.8 (C-6), 35.8 (C-7), 30.9 (C-8), 28.9 (C-9), 29.1 (C-10), 29.1 (C-11), 25.3 (C-12), 34.1 (C-13), 75.8 (C-14), 36.2 (C-15), 18.6 (C-16), 13.9 (C-17), 172.6 (C-1′), 104.9 (C-2′), 165.2 (C-3′), 101.4 (C-4′), 160.7 (C-5′), 111.1 (C-6′), 148.7 (C-7′), 36.5 (C-8′), 31.9 (C-9′), 29.5 (C-10′), 29.3 (C-11′), 29.2 (C-12′), 25.4 (C-13′), 34.1 (C-14′), 75.1 (C-15′), 36.5 (C-16′), 18.6 (C-17′), 14.0 (C-18′), 171.6 (C-1″), 21.4 (C-2″)。以上数据与文献对照[12-13]，鉴定化合物 **12** 为 integracin A。

化合物 **13**：黄色油状物，ESI-MS *m*/*z*: 587 $[M+H]^+$。^{1}H-NMR (500 MHz, $CDCl_3$) *δ*: 6.10 (1H, d, *J* = 7.4 Hz, H-1), 6.06 (1H, brs, H-3), 6.10 (1H, d, *J* = 2.4 Hz, H-5), 2.41 (1H, t, *J* = 8.0 Hz, H-7α), 1.50 (1H, m, H-8β), 1.22 (4H, m, H-9～12), 1.65 (1H, m, H-13), 5.23 (1H, m, H-14), 1.62 (1H, m, H-15), 1.30 (1H, m, H-16), 0.94 (1H, m, H-17), 6.19 (1H, d, *J* = 2.4 Hz, H-4′α), 6.20 (1H, d, *J* = 2.4 Hz, H-6′α), 2.81 (1H, m, H-8′β), 1. 50 (1H, m, H-9′β), 1.22 (4H, m, H-10′～13′), 1.45 (1H, m, H-14′), 3.49 (1H, m, H-15′), 1.45 (1H, m, H-16′), 1.35 (1H, m, H-17′), 0.90 (1H, m, H-18′α)；^{13}C-NMR (125 MHz, $CDCl_3$) *δ*: 107.9 (C-1), 159.2 (C-2), 100.9 (C-3), 159.2 (C-4), 107.9 (C-5), 146.3 (C-6), 36.9 (C-7), 32.4 (C-8), 30.3 (C-9), 30.4 (C-10), 30.5 (C-11), 26.7 (C-12), 35.5 (C-13), 76.6 (C-14), 37.8 (C-15), 19.9 (C-16), 14.3 (C-17), 172.8 (C-1′), 105.7 (C-2′), 166.1 (C-3′), 101.9 (C-4′), 163.5 (C-5′), 111.8 (C-6′), 149.1 (C-7′), 37.8 (C-8′), 33.6 (C-9′), 31.0 (C-10′), 30.9 (C-11′), 30.9 (C-12′), 26.8 (C-13′), 35.5 (C-14′), 72.2 (C-15′), 38.4 (C-16′), 19.9 (C-17′), 14.4 (C-18′)。以上数据与文献对照[13]，鉴定化合物 **13** 为 integracin B。

化合物 **14**：白色无定形粉末，ESI-MS *m*/*z*: 487

[M＋H]$^+$。^{1}H-NMR (500 MHz, $CDCl_3$) δ: 1.40 (1H, m, H-1α), 2.10 (1H, m, H-3β), 2.20 (1H, m, H-2α), 2.87 (1H, ddd, J = 15.0, 11.0, 4.1 Hz, H-2β), 4.45 (1H, m, H-6), 5.36 (1H, m, H-12), 1.58 (1H, ddd, J = 13.5, 4.5, 2.0 Hz, H-16α), 2.53 (1H, td, J = 13.5, 4.5 Hz, H-16β), 2.59 (1H, s, H-18), 1.22 (3H, s, H-23), 1.45 (3H, s, H-24), 1.56 (3H, s, H-25), 1.15 (3H, s, H-26), 1.20 (3H, s, H-27), 1.34 (3H, s, H-29), 0.95 (3H, d, J = 6.0 Hz, H-23)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 39.8 (C-1), 34.8 (C-2), 215.8 (C-3), 49.4 (C-4), 57.2 (C-5), 68.4 (C-6), 41.4 (C-7), 39.8 (C-8), 47.7 (C-9), 36.9 (C-10), 24.2 (C-11), 128.3 (C-12), 139.5 (C-13), 42.8 (C-14), 29.3 (C-15), 25.8 (C-16), 47.7 (C-17), 55.0 (C-18), 72.7 (C-19), 42.8 (C-20), 26.4 (C-21), 37.0 (C-22), 24.7 (C-23), 27.2 (C-24), 16.3 (C-25), 18.4 (C-26), 24.2 (C-27), 180.7 (C-28), 16.5 (C-30)。以上数据与文献对照[14]，鉴定化合物 **14** 为 6β,19α-二羟基乌苏-3-氧代-12-烯-28-酸。

化合物 **15**：白色无定形粉末，ESI-MS m/z: 457 [M＋H]$^+$。与乌苏酸对照品共薄层色谱，用 3 个不同体系展开剂展开，Rf 值和斑点颜色均一致。^{1}H-NMR (600 MHz, $CDCl_3$) δ: 4.26 (1H, s, H-3), 5.11 (1H, s, H-12), 0.75 (3H, s, H-23), 0.68 (3H, s, H-24), 0.89 (3H, s, H-25), 0.85 (3H, s, H-26), 1.04 (3H, s, H-27), 0.82 (3H, d, J = 6.1 Hz, H-29), 0.91 (3H, d, J = 6.0 Hz, H-30)；^{13}C-NMR (150 MHz, $CDCl_3$) δ: 38.5 (C-1), 27.6 (C-2), 77.7 (C-3), 39.1 (C-4), 55.2 (C-5), 18.6 (C-6), 33.2 (C-7), 39.5 (C-8), 47.8 (C-9), 39.9 (C-10), 23.6 (C-11), 122.8 (C-12), 139.1 (C-13), 42.6 (C-14), 27.3 (C-15), 24.6 (C-16), 47.5 (C-17), 53.1 (C-18), 39.5 (C-19), 39.5 (C-20), 31.2 (C-21), 37.6 (C-22), 28.1 (C-23), 15.9 (C-24), 16.0 (C-25), 16.8 (C-26), 23.5 (C-27), 179.1 (C-28), 16.8 (C-29), 21.4 (C-30)。以上数据与文献对照[15]，鉴定化合物 **15** 为乌苏酸。

化合物 **16**：白色无定形粉末，ESI-MS m/z: 457 [M＋H]$^+$。与齐墩果酸对照品共薄层色谱，用 3 个不同体系展开剂展开，Rf 值和斑点颜色均一致。^{1}H-NMR (600 MHz, $CDCl_3$) δ: 4.19 (1H, s, H-3), 5.15 (1H, s, H-12), 0.99 (3H, s, H-23), 0.71 (3H, s, H-24), 0.74 (3H, s, H-25), 0.89 (3H, s, H-26), 1.09 (3H, s, H-27), 0.85 (3H, s, H-29), 0.91 (3H, s, H-30)；^{13}C-NMR (150 MHz, $CDCl_3$) δ: 38.1 (C-1), 27.3 (C-2), 79.1 (C-3), 38.9 (C-4), 54.9 (C-5), 18.0 (C-6), 32.7 (C-7), 39.5 (C-8), 47.5 (C-9), 37.2 (C-10), 23.0 (C-11), 121.7 (C-12), 143.6 (C-13), 41.7 (C-14), 27.9 (C-15), 23.4 (C-16), 46.6 (C-17), 41.0 (C-18), 46.0 (C-19), 30.3 (C-20), 33.9 (C-21), 32.5 (C-22), 28.0 (C-23), 16.2 (C-24), 15.4 (C-25), 17.3 (C-26), 26.1 (C-27), 178.8 (C-28), 33.8 (C-29), 23.4 (C-30)。以上数据与文献对照[15]，鉴定化合物 **16** 为齐墩果酸。

化合物 **17**：白色针状结晶（醋酸乙酯），mp 136～137 ℃，ESI-MS m/z: 415 [M＋H]$^+$。与 β-谷甾醇对照品共薄层色谱，用 3 个不同体系展开剂展开，Rf 值和斑点颜色均一致。^{1}H-NMR (600 MHz, $CDCl_3$) δ: 3.52 (1H, m, H-3), 5.41 (1H, s, H-6), 0.67 (3H, s, H-18), 1.13 (3H, s, H-19), 1.01 (3H, d, J = 6.5 Hz, H-21), 0.81 (3H, d, J = 7.0 Hz, H-26), 0.82 (3H, d, J = 7.0 Hz, H-27), 0.84 (3H, d, J = 7.2 Hz, H-29)；^{13}C-NMR (150 MHz, $CDCl_3$) δ: 36.8 (C-1), 31.1 (C-2), 71.5 (C-3), 42.0 (C-4), 140.1 (C-5), 121.9 (C-6), 31.6 (C-7), 31.7 (C-8), 50.5 (C-9), 36.3 (C-10), 21.6 (C-11), 40.1 (C-12), 41.9 (C-13), 56.3 (C-14), 24.5 (C-15), 26.0 (C-16), 55.8 (C-17), 12.1 (C-18), 19.4 (C-19), 36.7 (C-20), 18.7 (C-21), 33.5 (C-22), 25.9 (C-23), 45.6 (C-24), 28.8 (C-25), 20.0 (C-26), 19.3 (C-27), 23.1 (C-28), 12.2 (C-29)。以上数据与文献对照[15]，鉴定化合物 **17** 为 β-谷甾醇。

化合物 **18**：白色针状结晶（醋酸乙酯），mp 290～292 ℃，ESI-MS m/z: 577 [M＋H]$^+$。与 β-胡萝卜苷对照品共薄层色谱，用 3 个不同体系展开剂展开，Rf 值和斑点颜色均一致。^{1}H-NMR (600 MHz, DMSO-d_6) δ: 5.30 (1H, d, J = 5.0 Hz, H-6), 0.64 (3H, s, H-18), 0.97 (3H, s, H-19), 0.90 (3H, d, J = 6.5 Hz, H-21), 0.80～0.82 (6H, d, J = 6.8 Hz, H-26, 27), 0.85 (3H, t, J = 7.2 Hz, H-29), 4.91 (1H, d, J = 6.5 Hz, H-1′), 3.62～3.66 (2H, m, H-6′)；^{13}C-NMR (150 MHz, DMSO-d_6) δ: 37.3 (C-1), 30.3 (C-2), 77.3 (C-3), 39.5 (C-4), 141.0 (C-5), 121.9 (C-6), 31.9 (C-7), 31.7 (C-8), 50.4 (C-9), 39.1 (C-10), 21.1 (C-11), 40.2 (C-12), 42.5 (C-13), 56.8 (C-14), 24.2 (C-15), 28.5 (C-16), 56.2 (C-17), 12.5 (C-18), 19.5 (C-19), 36.9 (C-20), 19.3 (C-21), 35.0 (C-22), 26.1 (C-23), 45.6 (C-24), 29.2 (C-25), 20.2 (C-26), 19.5 (C-27), 23.4 (C-28), 12.3 (C-29), 101.6 (C-1′), 74.5 (C-2′), 77.5 (C-3′), 71.9 (C-4′), 77.3 (C-5′), 62.7 (C-6′)。以上数据

与文献对照[16]，鉴定化合物 **18** 为 β-胡萝卜苷。

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