

银背风毛菊三萜成分研究

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摘要: 目的 研究银背风毛菊 *Saussurea nivea* 全草的化学成分。方法 应用硅胶色谱技术及重结晶等方法对化合物进行分离, 通过现代波谱技术鉴定化合物的结构。结果 从银背风毛菊丙酮提取物中分离了 11 个三萜类化合物, 分别鉴定为 20(30)-烯-3β-羟基蒲公英烷 (**1**)、20-烯-3β-羟基蒲公英烷 (**2**)、1β-hydroxy-ursa-9(11)、12(13)-dien-3β-yl palmitate (**3**)、1β-hydroxy-oleana-9(11)、12-dien-3β-yl palmitate (**4**)、21α-hydroxy-taraxasterol (**5**)、20(30)-烯-21α, 22α-环氧-3β-蒲公英醇 (**6**)、12-ursene-3β, 11α-diol 3-O-palmitate (**7**)、9(11)、12-ursadien-3β-ol 3-O-palmitate (**8**)、bauereny lacetate (**9**)、3β-hydroxy-11-oxo-olean-12-enyl palmitate (**10**)、3β-hydroxy-11α, 12α-epoxy-friedoolean-14-enyl palmitate (**11**)。结论 所有化合物均是首次从银背风毛菊中分离得到, 其中化合物 **7~11** 为首次从风毛菊属植物中分离得到。

关键词: 菊科; 银背风毛菊; 三萜; 20(30)-烯-3β-羟基蒲公英烷; 20-烯-3β-羟基蒲公英烷

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Triterpenoids from *Saussurea nivea*

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Key words: Asteraceae; *Saussurea nivea* Turcz.; triterpenoid; 3β-hydroxy-20(30)-taraxastene; 3β-hydroxyl-20-taraxastene

银背风毛菊 *Saussurea nivea* Turcz. 为菊科风毛菊属植物, 主要分布于东北及内蒙古自治区等地。民间用于治疗感冒、头痛、关节炎和扭伤^[1-2]。国内外对该植物的化学成分研究报道^[3]较少。为从银背风毛菊中寻找新的生物活性成分, 对采自河南省桐柏山的银背风毛菊全草进行了化学成分研究。应用色谱分离技术及重结晶等方法对化合物进行分离, 并运用现代波谱技术 (¹H-NMR、¹³C-NMR、EI-MS 等) 鉴定化合物结构。从其丙酮提取物中分离鉴定了 11 个三萜类化合物, 分别是 20(30)-烯-3β-羟基蒲公英烷 (蒲公英甾醇, taraxasterol, **1**)、20-烯-3β-羟基蒲公英烷 (pseudotaraxasterol, **2**)、1β-hydroxy-ursa-9(11)、12(13)-dien-3β-yl palmitate (ussuriensin A, **3**)、1β-hydroxy-oleana-9(11)、12-dien-3β-yl palmitate (ussuriensin B, **4**)、21α-hydroxyl-taraxasterol (**5**)、20(30)-烯-21α, 22α-环氧-3β-蒲公英醇 (ptiloexp-oxide, **6**)、12-ursene-3β, 11α-diol 3-O-palmitate (**7**)、9(11)、12-ursadien-3β-ol 3-O-

palmitate (**8**)、bauereny lacetate (**9**)、3β-hydroxy-11-oxo-olean-12-enyl palmitate (**10**)、3β-hydroxy-11α, 12α-epoxy-friedo-olean-14-enyl palmitate (**11**)。所有化合物均为首次从银背风毛菊中分离得到, 其中化合物 **7~11** 为首次从风毛菊属植物中分离得到。

1 仪器与材料

Bruker AM-400 核磁共振波谱仪(TMS 内标); Kofler 显微熔点仪; 色谱用硅胶 (200~300 目) 和薄层色谱 GF₂₅₄ 硅胶板为青岛海洋化工厂产品。溶剂均为分析纯, 为天津市红岩试剂厂产品。

银背风毛菊 *Saussurea nivea* Turcz. 全草于 2004 年 9 月采自河南省桐柏山, 由河南农业大学植物分类学专家朱长山教授鉴定, 标本 (2004056) 保存于新乡医学院药学院中药标本馆。

2 提取与分离

银背风毛菊全草阴干粉碎 (约 10 kg), 丙酮室温下浸泡提取 3 次, 每次 7 d, 减压浓缩得浸膏 230 g。将所得浸膏经硅胶柱色谱分离, 以石油醚-丙酮

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(50:1~1:1) 梯度洗脱, 得到 Fr. 1~7 部分。Fr. 1 部分经硅胶柱色谱分离, 以石油醚-醋酸乙酯梯度洗脱并重结晶得化合物 **7** (23 mg)、**8** (25 mg)、**9** (45 mg)、**10** (63 mg)、**11** (42 mg); Fr. 2 部分经硅胶柱色谱分离, 以石油醚-醋酸乙酯梯度洗脱并重结晶得化合物 **1** (125 mg)、**2** (92 mg)、**6** (26 mg); Fr. 4 部分经硅胶柱色谱分离, 以氯仿-丙酮梯度洗脱并重结晶得化合物 **3** (18 mg)、**4** (15 mg); Fr. 5 部分经硅胶柱色谱分离, 以石油醚-醋酸乙酯梯度洗脱并重结晶得化合物 **5** (25 mg)。

3 结构鉴定

化合物 1: 白色粉末(丙酮), mp 205~206 °C, Liebermann-Burchard 反应阳性。¹H-NMR (CDCl₃, 400 MHz) δ: 4.61, 4.59 (各 1H, d, J = 2.0 Hz, H-30), 3.20 (1H, dd, J = 11.2, 4.8 Hz, H-3α), 1.01 (3H, s, CH₃-26), 1.00 (3H, d, J = 6.8 Hz, CH₃-29), 0.96 (3H, s, CH₃-23), 0.92 (3H, s, CH₃-27), 0.84 (3H, s, CH₃-28), 0.84 (3H, s, CH₃-25), 0.74 (3H, s, CH₃-24); ¹³C-NMR (CDCl₃, 100 MHz) δ: 154.64 (C-20), 107.11 (C-30), 79.01 (C-3), 55.32 (C-5), 50.46 (C-9), 48.65 (C-18), 42.01 (C-14), 40.87 (C-8), 39.36 (C-19), 39.14 (C-13), 38.84 (C-4), 38.83 (C-22), 38.73 (C-1), 38.28 (C-16), 37.11 (C-10), 34.51 (C-17), 34.03 (C-7), 27.96 (C-23), 27.37 (C-2), 26.63 (C-15), 26.16 (C-12), 25.60 (C-21), 25.48 (C-29), 21.43 (C-11), 19.47 (C-28), 18.27 (C-6), 16.80 (C-25), 15.87 (C-26), 15.36 (C-24), 14.74 (C-27)。以上数据与文献数据基本一致^[4], 鉴定化合物 **1** 为 20(30)-烯-3β-羟基蒲公英烷。

化合物 2: 白色粉末(丙酮), mp 217~219 °C, Liebermann-Burchard 反应阳性。¹H-NMR (CDCl₃, 400 MHz) δ: 5.24 (1H, dd, J = 6.8, 1.9 Hz, H-21), 3.20 (1H, dd, J = 11.2, 4.8 Hz, H-3), 1.62 (3H, s, CH₃-30), 1.03 (3H, s, CH₃-26), 0.99 (3H, d, J = 6.0 Hz, CH₃-29), 0.97 (3H, s, CH₃-27), 0.94 (3H, s, CH₃-23), 0.84 (3H, s, CH₃-25), 0.75 (3H, s, CH₃-24), 0.72 (3H, s, CH₃-28); ¹³C-NMR (CDCl₃, 100 MHz) δ: 139.85 (C-20), 118.86 (C-21), 79.01 (C-3), 55.26 (C-5), 50.39 (C-9), 48.60 (C-18), 42.31 (C-14), 42.15 (C-22), 41.04 (C-8), 39.14 (C-13), 38.84 (C-4), 38.73 (C-1), 37.11 (C-10), 36.68 (C-16), 36.29 (C-19), 34.37 (C-17), 34.20 (C-7), 27.96 (C-23), 27.61 (C-12), 27.37 (C-2), 27.01 (C-15), 22.53 (C-29), 21.64 (C-30), 21.59 (C-11), 18.27 (C-6), 17.69 (C-28), 16.26 (C-25), 16.02

(C-26), 15.36 (C-24), 14.74 (C-27)。与文献数据基本一致^[4], 鉴定化合物 **2** 为 20-烯-3β-羟基蒲公英烷。

化合物 3: 针状晶体(甲醇), Liebermann-Burchard 反应阳性。¹H-NMR (CDCl₃, 400 MHz) δ: 6.51 (1H, d, J = 6.0 Hz, H-11), 5.22 (1H, d, J = 6.0 Hz, H-12), 4.30 (1H, dd, J = 8.2, 3.4 Hz, H-3), 3.55 (1H, dd, J = 11.6, 4.8 Hz, H-1), 2.28 (2H, t, J = 7.4 Hz, H-2'), 1.26 (br s, H-4'~15'), 0.96 (3H, s, CH₃-28), 0.91 (3H, s, CH₃-27), 0.88 (3H, s, CH₃-25), 0.87 (3H, t, J = 8.0 Hz, H-16'), 0.87 (3H, s, CH₃-24), 0.84 (3H, s, CH₃-23), 0.83 (3H, d, J = 6.4 Hz, CH₃-29), 0.80 (3H, d, J = 6.4 Hz, CH₃-30), 0.78 (3H, s, CH₃-26); ¹³C-NMR (CDCl₃, 100 MHz) δ: 173.61 (C-1'), 152.07 (C-9), 141.55 (C-13), 123.49 (C-12), 117.10 (C-11), 76.86 (C-3), 75.55 (C-1), 57.21 (C-18), 48.78 (C-5), 44.10 (C-10), 43.20 (C-8), 41.30 (C-22), 40.79 (C-14), 39.39 (C-19), 39.04 (C-20), 37.98 (C-4), 34.73 (C-2), 34.51 (C-2'), 33.68 (C-17), 31.91 (C-14'), 31.19 (C-21), 30.98 (C-7), 29.36~29.68 (C-4'~13'), 28.67 (C-23), 28.23 (C-16), 27.70 (C-28), 26.19 (C-15), 25.12 (C-3'), 22.90 (C-15'), 22.68 (C-27), 21.51 (C-30), 18.66 (C-25), 18.28 (C-6), 17.80 (C-26), 17.42 (C-29), 16.24 (C-24), 14.12 (C-16')。与文献数据基本一致^[2], 鉴定化合物 **3** 为 1β-hydroxyursa-9(11), 12(13)-dien-3β-yl palmitate。

化合物 4: 针状晶体(甲醇), Liebermann-Burchard 反应阳性。¹H-NMR (CDCl₃, 400 MHz) δ: 6.51 (1H, d, J = 6.0 Hz, H-11), 5.51 (1H, d, J = 6.4 Hz, H-12), 4.54 (1H, dd, J = 12.0, 4.4 Hz, H-3), 3.91 (1H, dd, J = 11.6, 4.8 Hz, H-1), 2.32 (2H, t, J = 7.4 Hz, H-2'), 1.26 (br s, H-4'~15'), 1.11 (3H, s, CH₃-27), 0.98 (3H, s, CH₃-30), 0.88 (3H, s, CH₃-24), 0.87 (3H, t, J = 7.6 Hz, H-16'), 0.87 (6H, s, CH₃-25, CH₃-28), 0.86 (3H, s, CH₃-23), 0.85 (3H, s, CH₃-29), 0.84 (3H, s, CH₃-26); ¹³C-NMR (CDCl₃, 100 MHz) δ: 173.53 (C-1'), 151.86 (C-9), 147.40 (C-13), 121.09 (C-12), 118.28 (C-11), 76.68 (C-3), 75.59 (C-1), 48.92 (C-5), 46.72 (C-19), 45.49 (C-18), 44.71 (C-10), 42.85 (C-14), 40.73 (C-8), 37.99 (C-4), 36.99 (C-22), 34.73 (C-2), 34.60 (C-2'), 34.60 (C-21), 33.18 (C-29), 32.14 (C-17), 31.92 (C-14'), 31.20 (C-7), 31.09 (C-20), 29.15~29.68 (C-4'~13'), 28.68 (C-23), 27.75 (C-28), 27.18 (C-16), 25.69 (C-15), 25.14 (C-3'), 23.67 (C-27),

22.69 (C-15'), 21.69 (C-30), 20.25 (C-26), 18.57 (C-25), 18.23 (C-6), 16.26 (C-24), 14.13 (C-16')。以上数据与文献数据基本一致^[2], 鉴定化合物 4 为 1 β -hydroxy-oleana-9(11), 12-dien-3 β -yl palmitate。

化合物 5: 针状晶体(甲醇), Liebermann-Burchard 反应阳性。¹H-NMR (C₅D₅N, 400 MHz) δ : 5.11, 5.01 (各 1H, d, J = 2.0 Hz, H-30), 4.72 (1H, m, H-21), 3.48 (1H, dd, J = 9.2, 6.4 Hz, H-3), 2.28 (1H, dq, J = 7.2 Hz, H-19), 2.14 (1H, dd, J = 13.8, 9.0 Hz, H-18), 1.50 (3H, d, J = 6.8 Hz, CH₃-29), 1.25 (3H, s, CH₃-26), 1.06 (3H, s, CH₃-23), 1.02 (3H, s, CH₃-27), 0.94 (3H, s, CH₃-25), 0.91 (3H, s, CH₃-24), 0.90 (3H, s, CH₃-28); ¹³C-NMR (C₅D₅N, 100 MHz) δ : 157.90 (C-20), 112.00 (C-30), 78.13 (C-3), 70.58 (C-21), 55.91 (C-5), 50.74 (C-9), 50.65 (C-22), 48.29 (C-18), 42.46 (C-14), 41.25 (C-8), 39.58 (C-4), 39.43 (C-13), 39.30 (C-1), 39.00 (C-19), 38.54 (C-16), 37.46 (C-10), 34.52 (C-7), 32.38 (C-17), 28.72 (C-29), 28.38 (C-2), 28.21 (C-23), 26.84 (C-15), 26.81 (C-12), 21.71 (C-11), 19.12 (C-28), 18.81 (C-6), 16.64 (C-25), 16.45 (C-26), 16.18 (C-24), 14.98 (C-27)。以上数据与文献数据基本一致^[5], 鉴定化合物 5 为 21 α -hydroxy-taraxasterol。

化合物 6: 白色粉末(丙酮), mp 219~221 °C, Liebermann-Burchard 反应阳性。¹H-NMR (CDCl₃, 400 MHz) δ : 5.04, 4.85 (各 1H, br s, H-30), 3.45 (1H, d, J = 4.8 Hz, H-21), 2.89 (1H, d, J = 4.8 Hz, H-22), 3.19 (1H, dd, J = 11.2, 5.2 Hz, H-3), 1.99 (1H, dq, J = 7.0 Hz, H-19), 1.03 (3H, d, J = 6.8 Hz, CH₃-29), 1.00 (3H, s, CH₃-26), 0.95 (3H, s, CH₃-23), 0.93 (3H, s, CH₃-27), 0.82 (3H, s, CH₃-25), 0.79 (3H, s, CH₃-28), 0.74 (3H, s, CH₃-24); ¹³C-NMR (CDCl₃, 100 MHz) δ : 151.31 (C-20), 111.98 (C-30), 78.91 (C-3), 63.98 (C-22), 56.06 (C-21), 55.23 (C-5), 50.34 (C-9), 42.20 (C-14), 42.11 (C-18), 40.95 (C-8), 38.83 (C-4), 38.69 (C-1), 37.89 (C-13), 37.07 (C-10), 36.26 (C-17), 36.12 (C-19), 34.07 (C-7), 33.60 (C-16), 27.96 (C-23), 27.33 (C-2), 27.22 (C-29), 26.49 (C-15), 26.17 (C-12), 21.39 (C-11), 18.24 (C-6), 16.23 (C-25), 15.94 (C-26), 15.33 (C-24), 15.10 (C-28), 14.78 (C-27)。以上数据与文献数据基本一致^[6], 鉴定化合物 6 为 20(30)-烯-21 α , 22 α -环氧-3 β -蒲公英醇。

化合物 7: 片状晶体(甲醇), Liebermann-Burchard 反应阳性。¹H-NMR (CDCl₃, 400 MHz) δ :

5.16 (1H, d, J = 3.2 Hz, H-12), 4.50 (1H, dd, J = 10.0, 4.0 Hz, H-3), 4.22 (1H, m, H-11), 1.23 (br s, H-4'~13'), 1.13 (3H, s, CH₃-27), 1.09 (3H, s, CH₃-25), 1.03 (3H, s, CH₃-26), 0.90 (3H, br s, CH₃-30), 0.86 (3H, s, CH₃-24), 0.86 (3H, s, CH₃-23), 0.85 (3H, s, CH₃-28), 0.84 (3H, d, J = 6.2 Hz, CH₃-29), 0.83 (3H, t, J = 6.8 Hz, CH₃-16'); ¹³C-NMR (CDCl₃, 100 MHz) δ : 173.60 (C-1'), 142.76 (C-13), 128.75 (C-12), 80.30 (C-3), 68.29 (C-11), 58.09 (C-18), 55.64 (C-9), 55.37 (C-5), 43.24 (C-14), 42.08 (C-8), 41.28 (C-22), 40.42 (C-1), 39.39 (C-20), 39.27 (C-19), 37.98 (C-4), 37.93 (C-10), 34.83 (C-2'), 33.59 (C-17), 33.59 (C-7), 31.91 (C-14'), 31.06 (C-21), 29.17~29.66 (C-4'~13'), 28.65 (C-28), 28.21 (C-23), 27.89 (C-16), 26.64 (C-15), 25.14 (C-3'), 23.75 (C-2), 23.09 (C-27), 22.67 (C-15'), 21.31 (C-30), 18.21 (C-6), 17.94 (C-26), 17.54 (C-29), 16.83 (C-25), 16.72 (C-24), 14.11 (C-16')。以上数据与文献数据基本一致^[7], 鉴定化合物 7 为 12-ursene-3 β , 11 α -diol 3-O-palmitate。

化合物 8: 片状晶体(甲醇), Liebermann-Burchard 反应阳性。¹H-NMR (CDCl₃, 400 MHz) δ : 5.56 (1H, d, J = 6.0 Hz, H-11), 5.42 (1H, d, J = 6.0 Hz, H-12), 4.50 (1H, dd, J = 10.0, 4.0 Hz, H-3), 1.23 (br s, H-4'~13'), 1.21 (3H, s, CH₃-25), 1.15 (3H, s, CH₃-26), 0.90 (3H, br s, CH₃-30), 0.86 (3H, s, CH₃-23), 0.86 (3H, s, CH₃-24), 0.87 (3H, br s, CH₃-29), 0.85 (3H, s, CH₃-27), 0.85 (3H, s, CH₃-28), 0.84 (3H, t, J = 6.5 Hz, CH₃-16'); ¹³C-NMR (CDCl₃, 100 MHz) δ : 173.60 (C-1'), 154.14 (C-9), 141.29 (C-13), 122.94 (C-12), 115.47 (C-11), 80.22 (C-3), 57.26 (C-18), 51.18 (C-5), 43.05 (C-8), 41.28 (C-22), 40.62 (C-14), 39.39 (C-20), 38.96 (C-19), 38.50 (C-4), 37.93 (C-10), 34.83 (C-2'), 33.59 (C-17), 31.91 (C-7), 31.91 (C14'), 31.06 (C-21), 29.20~29.67 (C-4'~13'), 28.65 (C-28), 28.20 (C-15), 26.06 (C-16), 25.42 (C-27), 25.14 (C-3'), 24.25 (C-2), 22.67 (C-15'), 22.08 (C-26), 21.50 (C-30), 18.21 (C-6), 17.54 (C-25), 17.40 (C-24), 16.83 (C-29), 14.11 (C-16')。以上数据与文献数据基本一致^[7], 鉴定化合物 8 为 12-ursadien-3 β -ol 3-O-palmitate。

化合物 9: 白色固体(丙酮), Liebermann-Burchard 反应阳性。¹H-NMR (CDCl₃, 400 MHz) δ : 5.40 (1H, d, J = 3.2 Hz, H-7), 4.50 (1H, dd, J = 11.0, 4.2 Hz, H-3), 2.04 (3H, s, CH₃-2'), 1.02, 0.98, 0.93,

0.91, 0.83, 0.75, (各 3H, s, CH₃-23~28), 1.03 (3H, d, *J* = 7.2 Hz, CH₃-30), 0.89 (3H, d, *J* = 6.0 Hz, CH₃-29); ¹³C-NMR (CDCl₃, 100 MHz) δ: 171.01 (C-1'), 145.43 (C-8), 116.22 (C-7), 81.12 (C-3), 54.85 (C-18), 50.52 (C-5), 48.10 (C-9), 41.23 (C-14), 37.99 (C-20), 37.80 (C-13), 37.70 (C-4), 37.66 (C-22), 36.49 (C-1), 35.28 (C-19), 35.05 (C-10), 32.33 (C-12), 32.02 (C-17), 32.02 (C-28), 31.49 (C-16), 29.18 (C-21), 28.84 (C-15), 27.49 (C-23), 25.63 (C-29), 24.17 (C-2), 23.94 (C-6), 23.65 (C-26), 22.65 (C-27), 22.54 (C-30), 21.35 (C-2'), 16.79 (C-11), 15.79 (C-24), 13.01 (C-25)。以上数据与文献数据基本一致^[8], 鉴定化合物 9 为 bauereny lacetate。

化合物 10: 白色油脂状(丙酮), Liebermann-Burchard 反应阳性。¹H-NMR (CDCl₃, 400 MHz) δ: 5.54 (1H, s, H-12), 4.49 (1H, m, H-3), 1.21 (br s, H-4'~13'), 1.32, 1.12, 1.09, 0.86, 0.85, 0.85, 0.84, 0.82 (各 3H, s, CH₃-23~30); ¹³C-NMR (CDCl₃, 100 MHz) δ: 199.97 (C-11), 173.54 (C-1'), 170.80 (C-13), 128.03 (C-12), 80.18 (C-3), 61.61 (C-9), 54.97 (C-5), 47.55 (C-18), 45.37 (C-14), 45.10 (C-19), 43.32 (C-8), 38.73 (C-1), 38.03 (C-4), 36.88 (C-10), 36.47 (C-22), 34.78 (C-2'), 34.40 (C-21), 33.04 (C-29), 32.65 (C-7), 32.32 (C-17), 31.91 (C-14'), 31.00 (C-20), 29.69~29.16 (C-4'~13'), 28.72 (C-28), 28.03 (C-23), 26.40 (C-16), 26.35 (C-15), 25.12 (C-3'), 23.56 (C-2), 23.46 (C-30), 23.36 (C-27), 22.68 (C-15'), 18.66 (C-26), 17.35 (C-6), 16.97 (C-25), 16.64 (C-24), 14.13 (C-16')。以上数据与文献数据基本一致^[9], 鉴定化合物 10 为 3β-hydroxy-11-oxo-olean-12-enyl palmitate。

化合物 11: 白色油脂状(丙酮), Liebermann-Burchard 反应阳性。¹H-NMR (CDCl₃, 400 MHz) δ: 5.54 (1H, dd, *J* = 8.4, 3.2 Hz, H-15), 4.50 (1H, dd, *J* = 12.0, 6.0 Hz, H-3), 3.10 (1H, t, *J* = 5.0 Hz, H-11), 2.79 (1H, d, *J* = 5.0 Hz, H-12), 1.09, 1.07, 0.99, 0.95, 0.89, 0.86, 0.85, 0.81 (各 3H, s, CH₃-23~30); ¹³C-NMR (CDCl₃, 100 MHz) δ: 173.50 (C-1'), 156.99 (C-14),

118.88 (C-15), 80.28 (C-3), 58.16 (C-12), 54.58 (C-5), 53.42 (C-11), 51.86 (C-9), 48.04 (C-18), 40.17 (C-19), 38.88 (C-8), 38.17 (C-22), 37.85 (C-1), 37.65 (C-4), 37.44 (C-10), 36.51 (C-21), 36.47 (C-13), 35.35 (C-17), 35.19 (C-16), 34.80 (C-2'), 33.64 (C-29), 33.10 (C-7), 31.9 (C-14'), 30.21 (C-27), 29.91 (C-28), 29.69~29.19 (C-4'~13'), 28.69 (C-20), 27.88 (C-23), 27.01 (C-26), 25.14 (C-3'), 23.22 (C-2), 22.69 (C-15'), 19.50 (C-30), 18.75 (C-6), 16.99 (C-24), 16.66 (C-25), 14.31 (C-16')。以上数据与文献数据基本一致^[9], 鉴定化合物 11 为 3β-hydroxy-11α, 12α-epoxy-friedoolean-14-enyl palmitate。

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