

川楝树皮乙酸乙酯提取物中甘遂烷型三萜类化学成分

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摘要: 从川楝 (*Melia azedarach* Linn.) 树皮乙酸乙酯提取物共鉴定到 10 种甘遂烷型三萜类化合物, 即苦内酯 (1)、3 α -hydroxytirucalla-7, 24 (25)-dien-6-oxo-21, 16-olide (2)、meliassenin B (3)、sendanolactone (4)、12 β -hydroxykulactone (5)、mesendanin O (6)、6 β -hydroxykulactone (7)、meliassenin L (8)、meliastatin 5 (9) 和 toosendine G (10), 其中化合物 2 和 5 首次从川楝中分离得到。

关键词: 川楝树皮; 乙酸乙酯提取物; 甘遂烷型三萜类; 化学成分

中图分类号: Q946.8; R284.1 文献标志码: A 文章编号: 1674-7895(2023)05-0089-03

DOI: 10.3969/j.issn.1674-7895.2023.05.09

Chemical components of tirucallane-type triterpenoids in ethyl acetate extract from the barks of *Melia azedarach*
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Abstract: Ten compounds of tirucallane-type triterpenoids were identified from the ethyl acetate extract from the barks of *Melia azedarach* Linn., namely kulactone (1), 3 α -hydroxytirucalla-7, 24 (25)-dien-6-oxo-21, 16-olide (2), meliassenin B (3), sendanolactone (4), 12 β -hydroxykulactone (5), mesendanin O (6), 6 β -hydroxykulactone (7), meliassenin L (8), meliastatin 5 (9), and toosendine G (10), among which compound 2 and 5 are isolated from *M. azedarach* for the first time.

Key words: bark of *Melia azedarach* Linn.; ethyl acetate extract; tirucallane-type triterpenoids; chemical component

川楝 (*Melia azedarach* Linn.) 为楝科 (Meliaceae) 楝属 (*Melia* Linn.) 植物, 主要分布于四川、贵州和云南等地, 具有行气止痛和驱虫等功效^[1]。川楝的干燥根皮及树皮是传统中药苦楝皮的重要来源, 但有关川楝树皮化学成分分离纯化的研究尚不全面, 川楝树皮中化学成分结构类型及其物质活性有待进一步探索。鉴于此, 本文对川楝树皮乙酸乙酯提取物开展了分离纯化研究, 以期对川楝树皮的进一步开发和利用提供基础数据。

1 材料和方法

1.1 材料

于 2019 年 9 月, 在贵州省兴义市南盘江镇采集株龄 5 a 川楝的树皮, 晒干后保存、备用。标本由贵州黔西南喀斯特区域发展研究院的邓超义教授鉴定。

1.2 方法

1.2.1 化合物分离 将晒干的川楝树皮样品 (23 kg) 粉碎成粗粉, 用体积分数 95% 乙醇加热回流提取 2 次, 每次 2 h, 减压浓缩得到浸膏, 浸膏用乙酸乙酯 (分析纯) 萃取, 得到乙酸乙酯部位浸膏 (800 g)。对乙酸乙酯部位浸膏用聚酰胺拌样, 用体积分数

40%~100% 甲醇 (分析纯) 溶液梯度洗脱, 分离得到 6 个流分 (Fr. A~F), 然后依次通过 C₁₈ 反相柱层析、硅胶柱层析、凝胶柱层析以及高效液相色谱仪纯化后得到化合物 1 (500.0 mg)、化合物 2 (2.6 mg)、化合物 3 (31.8 mg)、化合物 4 (119.8 mg)、化合物 5 (9.2 mg)、化合物 6 (8.9 mg)、化合物 7 (11.1 mg)、化合物 8 (3.7 mg)、化合物 9 (2.4 mg) 和化合物 10 (4.0 mg)。

1.2.2 高效液相条件 Agilent 色谱柱 (250 mm×9.4 mm, 5 μ m), 柱温 28 $^{\circ}$ C, 流动相为体积分数 85%~45% 的甲醇-水溶液, 流速 2 mL·min⁻¹, 检测波长为 210、230、254 和 280 nm。

2 结果和分析

化合物 1: 无色晶体, 熔点 163 $^{\circ}$ C~164 $^{\circ}$ C, 分子式 C₃₀H₄₄O₃, ESI-MS m/z : 475 [M+Na]⁺。¹H-NMR (600 MHz, CDCl₃) δ_{H} : 5.30 (1H, d, J =3.0 Hz, H-7), 5.06 (1H, m, H-24), 4.12 (1H, m, H-16), 2.72 (2H, m, H-2), 2.46 (1H, m, H-9), 2.38 (1H, ddd, J =12.5, 8.1, 4.4 Hz, H-20), 2.25 (2H, m, H-15), 2.21 (2H, m, H-6), 2.11 (2H, d, J =3.9 Hz, H-11), 1.99 (2H, m, H-23), 1.92 (2H, dd, J =11.3, 6.3, 3.7 Hz, H-12), 1.75 (2H, m, H-22), 1.69 (1H, m, H-5), 1.65 (3H, s,

收稿日期: 2023-04-27

基金项目: 国家自然科学基金项目 (32160102); 贵州省科技支撑计划 (黔科合基础-ZK[2021]一般 558; 黔科合基础-ZK[2021]一般 512)

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引用格式: 董晶晶, 邵莉莉, 李金玉, 等. 川楝树皮乙酸乙酯提取物中甘遂烷型三萜类化学成分[J]. 植物资源与环境学报, 2023, 32(5): 89-91.

H-27), 1.57(3H, s, H-26), 1.42(2H, m, H-1), 1.21(3H, s, H-30), 1.08(3H, s, H-29), 1.01(3H, s, H-28), 0.99(3H, s, H-18), 0.92(3H, s, H-19)。¹³C-NMR(150 MHz, CDCl₃) δ_C: 216.1(C-3), 180.5(C-21), 143.3(C-8), 132.5(C-25), 123.3(C-24), 118.3(C-7), 82.3(C-16), 58.0(C-17), 55.0(C-14), 52.4(C-5), 47.7(C-9), 47.7(C-4), 45.3(C-20), 39.4(C-13), 38.1(C-1), 35.5(C-15), 35.3(C-10), 34.6(C-2), 32.1(C-30), 29.5(C-22), 29.1(C-12), 25.9(C-6), 25.6(C-27), 24.3(C-11), 24.2(C-28), 21.4(C-29), 21.3(C-19), 17.8(C-26), 16.7(C-23), 12.3(C-18)。与文献[2]比对, 鉴定化合物1为苦内酯(kulactone)。

化合物2: 无色晶体, 熔点 203 °C ~ 204 °C, 分子式 C₃₀H₄₄O₄, ESI-MS *m/z*: 491 [M+Na]⁺。¹H-NMR(600 MHz, CDCl₃) δ_H: 5.68(1H, d, *J*=2.8 Hz, H-7), 5.09(1H, m, H-24), 4.14(1H, m, H-16), 3.35(1H, s, H-3), 3.03(1H, m, H-9), 2.57(1H, s, H-5), 2.43(2H, m, H-20), 2.32, 1.79(2H, m, H-15), 2.13(1H, m, H-17), 2.11, 2.03(2H, m, H-23), 1.96, 1.94(2H, m, H-11), 1.92, 1.86(2H, m, H-12), 1.87, 1.78(2H, m, H-2), 1.82, 1.34(2H, m, H-1), 1.68(3H, s, H-27), 1.61(3H, s, H-26), 1.48(3H, m, H-22), 1.26(3H, s, H-30), 1.25(3H, s, H-28), 1.13(3H, s, H-29), 0.97(3H, s, H-18), 0.86(3H, s, H-19)。¹³C-NMR(150 MHz, CDCl₃) δ_C: 200.8(C-6), 180.1(C-21), 167.3(C-8), 132.8(C-25), 124.6(C-7), 123.2(C-24), 81.6(C-16), 76.5(C-3), 60.7(C-5), 57.6(C-17), 55.9(C-14), 49.7(C-9), 45.2(C-20), 44.1(C-10), 39.2(C-4), 36.7(C-13), 34.8(C-15), 30.9(C-1), 29.6(C-30), 29.1(C-22), 29.0(C-12), 27.9(C-28), 26.0(C-23), 25.6(C-27), 24.4(C-2), 21.4(C-29), 21.3(C-18), 17.9(C-26), 16.4(C-11), 14.0(C-19)。与文献[3]比对, 鉴定化合物2为 3α-hydroxytirucalla-7,24(25)-dien-6-oxo-21,16-olide。

化合物3: 白色粉末, 分子式 C₃₀H₄₄O₄, ESI-MS *m/z*: 491 [M+Na]⁺。¹H-NMR(600 MHz, CDCl₃) δ_H: 5.70(1H, d, *J*=2.8 Hz, H-7), 5.09(1H, t, *J*=7.1, 1.5 Hz, H-24), 4.15(1H, ddd, *J*=11.3, 10.1, 7.7 Hz, H-16), 3.45(1H, s, H-5), 3.20(1H, dd, *J*=11.7, 3.6 Hz, H-3), 2.90(1H, m, H-9), 2.44(1H, ddd, *J*=12.5, 7.9, 4.6 Hz, H-20), 2.32, 1.76(2H, dd, *J*=13.8, 10.1 Hz, H-15), 2.12(1H, m, H-17), 2.03(2H, m, H-23), 1.94, 1.48(2H, m, H-22), 1.92, 1.64(2H, m, H-11), 1.82(2H, m, H-12), 1.68(3H, s, H-26), 1.66, 1.39(2H, m, H-1), 1.66, 1.57(2H, m, H-2), 1.61(3H, s, H-27), 1.30(3H, s, H-28), 1.26(3H, s, H-30), 1.10(3H, s, H-29), 0.98(3H, s, H-18), 0.85(3H, s, H-19)。¹³C-NMR(150 MHz, CDCl₃) δ_C: 199.2(C-6), 180.0(C-21), 167.2(C-8), 132.7(C-25), 124.6(C-7), 123.1(C-24), 81.5(C-16), 78.7(C-3), 65.6(C-5), 57.5(C-17), 55.8(C-14), 49.8(C-9), 45.1(C-20), 44.3(C-10), 39.2(C-13), 37.9(C-4), 36.6(C-1), 34.7(C-15), 29.5(C-30), 29.0(C-22), 28.9(C-12), 28.2(C-28), 26.3(C-2), 25.9(C-23), 25.6(C-26), 21.3(C-18), 17.8(C-27), 16.4(C-11), 14.7(C-29), 13.8(C-19)。与文献[4]比对, 鉴定化合物3为 meliasenin B。

化合物4: 白色粉末, 分子式 C₃₀H₄₂O₄, ESI-MS *m/z*: 489 [M+Na]⁺。¹H-NMR(600 MHz, CDCl₃) δ_H: 5.73(1H, t, *J*=2.4 Hz, H-7), 5.05(1H, t, *J*=5.4 Hz, H-24), 4.13(1H, m, H-16), 2.94(1H, ddd, *J*=11.3, 7.8, 2.8 Hz, H-9), 2.71, 2.28(2H, m, H-2), 2.70(1H, m, H-

20), 2.41(1H, s, H-5), 2.32, 1.89(2H, m, H-15), 2.13(1H, d, *J*=12.0 Hz, H-17), 2.06, 2.00(2H, m, H-23), 1.95, 1.70(2H, m, H-1), 1.94, 1.45(2H, m, H-22), 1.91, 1.75(2H, m, H-11), 1.82, 1.66(2H, m, H-12), 1.64(3H, s, H-27), 1.57(3H, s, H-26), 1.31(3H, s, H-29), 1.29(3H, s, H-28), 1.29(3H, s, H-30), 1.07(3H, s, H-19), 0.95(3H, s, H-18)。¹³C-NMR(150 MHz, CDCl₃) δ_C: 214.0(C-3), 197.6(C-6), 179.7(C-21), 167.5(C-8), 132.6(C-25), 124.3(C-7), 123.0(C-24), 81.2(C-16), 65.3(C-5), 57.4(C-17), 55.8(C-14), 48.9(C-9), 46.8(C-4), 44.9(C-20), 43.6(C-10), 39.1(C-13), 37.0(C-1), 34.6(C-15), 33.7(C-2), 29.4(C-30), 28.9(C-22), 28.7(C-12), 25.8(C-23), 25.5(C-27), 24.9(C-28), 21.4(C-29), 21.1(C-18), 17.7(C-26), 16.4(C-11), 13.3(C-19)。与文献[5]比对, 鉴定化合物4为 sendanolactone。

化合物5: 无色油状, 分子式 C₃₀H₄₂O₄, ESI-MS *m/z*: 491 [M+Na]⁺。¹H-NMR(600 MHz, CDCl₃) δ_H: 5.38(1H, d, *J*=3.0 Hz, H-7), 5.10(1H, t, *J*=6.6 Hz, H-24), 4.18(2H, ddd, *J*=11.6, 10.2, 7.3 Hz, H-16), 4.02(2H, dd, *J*=9.4, 5.4 Hz, H-12), 2.77(2H, td, *J*=14.6, 5.6 Hz, H-2), 2.52(1H, dd, *J*=12.6, 11.4 Hz, H-17), 2.28(2H, m, H-23), 1.99(2H, m, H-1), 1.73(1H, m, H-5), 1.69(3H, s, H-26), 1.62(3H, s, H-27), 1.38(3H, s, H-30), 1.12(3H, s, H-28), 1.05(3H, s, H-29), 1.04(3H, s, H-19), 0.83(3H, s, H-18)。¹³C-NMR(150 MHz, CDCl₃) δ_C: 216.1(C-3), 180.4(C-21), 143.0(C-8), 132.7(C-25), 123.6(C-24), 119.2(C-7), 82.1(C-16), 72.0(C-12), 54.9(C-13), 53.2(C-17), 52.4(C-5), 48.0(C-9), 47.8(C-4), 45.5(C-20), 44.4(C-14), 38.4(C-1), 36.3(C-15), 35.3(C-10), 34.7(C-2), 33.8(C-30), 30.1(C-11), 29.1(C-22), 26.0(C-23), 25.7(C-26), 24.4(C-29), 24.3(C-6), 21.4(C-28), 19.9(C-27), 17.9(C-18), 12.6(C-19)。与文献[6]比对, 鉴定化合物5为 12β-hydroxykulactone。

化合物6: 白色粉末, 分子式 C₃₀H₄₆O₄, ESI-MS *m/z*: 493 [M+Na]⁺。¹H-NMR(600 MHz, CDCl₃) δ_H: 5.35(1H, d, *J*=3.0 Hz, H-7), 5.13(1H, t, *J*=6.6 Hz, H-24), 4.20(1H, ddd, *J*=11.4, 10.3, 7.3 Hz, H-16), 4.03(2H, dt, *J*=9.7, 4.9 Hz, H-12), 2.54(1H, dd, *J*=12.6, 11.4 Hz, H-17), 2.47(1H, ddt, *J*=12.8, 6.5, 3.0 Hz, H-9), 2.40(2H, m, H-20), 2.38, 1.42(2H, m, H-11), 2.29, 1.74(2H, dd, *J*=13.7, 10.2 Hz, H-15), 2.20, 2.13(2H, m, H-23), 2.12, 1.99(2H, m, H-6), 1.96, 1.50(2H, m, H-22), 1.93, 1.62(2H, m, H-2), 1.82(1H, dd, *J*=12.2, 5.8 Hz, H-5), 1.71(2H, d, *J*=1.6 Hz, H-26), 1.65(3H, d, *J*=1.3 Hz, H-27), 1.59, 1.40(2H, m, H-1), 1.37(3H, s, H-30), 0.97(3H, s, H-28), 0.94(3H, s, H-29), 0.86(3H, s, H-18), 0.82(3H, s, H-19)。¹³C-NMR(150 MHz, CDCl₃) δ_C: 180.6(C-21), 143.0(C-8), 132.7(C-25), 123.7(C-24), 119.3(C-7), 82.2(C-16), 76.0(C-3), 72.2(C-12), 54.9(C-14), 53.2(C-17), 48.2(C-9), 45.5(C-20), 44.6(C-5), 44.4(C-13), 37.4(C-4), 36.2(C-15), 35.1(C-10), 33.6(C-30), 31.1(C-1), 30.3(C-11), 29.1(C-22), 27.7(C-28), 26.0(C-23), 25.7(C-26), 25.3(C-2), 23.9(C-6), 21.6(C-29), 19.8(C-18), 17.9(C-27), 12.8(C-19)。与文献[7]比对, 鉴定化合物6为 mesendanin O。

化合物7: 无色油状, 分子式 C₃₀H₄₆O₄, ESI-MS *m/z*: 491 [M+Na]⁺。¹H-NMR(600 MHz, CDCl₃) δ_H: 5.48(1H, dd, *J*=4.2, 2.8 Hz,

H-7), 5.12(1H, m, H-24), 4.50(1H, m, H-6), 4.17(1H, ddd, $J=11.4, 10.1, 7.6$ Hz, H-16), 2.88(2H, td, $J=14.1, 5.5$ Hz, H-2), 2.32, 1.77(2H, m, H-15), 2.00(1H, m, H-17), 1.71(3H, d, $J=1.4$ Hz, H-26), 1.64(3H, s, H-27), 1.54(3H, s, H-28), 1.51(1H, dd, $J=4.3, 2.6$ Hz, H-5), 1.33(3H, s, H-30), 1.28(3H, s, H-29), 1.25(3H, s, H-19), 0.94(3H, s, H-18)。 $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ_{C} : 215.8(C-3), 180.4(C-21), 146.0(C-8), 132.8(C-25), 123.3(C-24), 122.0(C-7), 82.1(C-16), 67.0(C-6), 58.0(C-17), 56.7(C-5), 55.1(C-14), 49.0(C-4), 48.8(C-9), 45.4(C-20), 40.0(C-1), 39.4(C-13), 35.5(C-10), 35.4(C-15), 34.6(C-2), 31.4(C-30), 29.5(C-22), 29.2(C-12), 26.0(C-23), 25.7(C-26), 24.7(C-28), 23.9(C-29), 21.4(C-18), 17.9(C-27), 16.8(C-11), 15.0(C-19)。与文献[8]比对, 鉴定化合物7为 β -hydroxykulactone。

化合物8: 无色粉末, 分子式 $\text{C}_{30}\text{H}_{48}\text{O}_4$, ESI-MS m/z : 495 [M+Na] $^+$ 。 $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 5.31(1H, d, $J=3.0$ Hz, H-7), 5.05(2H, brs, H-26), 4.30(1H, m, H-24), 4.06(1H, m, H-16), 2.78, 2.21(2H, td, $J=14.4, 5.4$ Hz, H-2), 2.27(1H, m, H-9), 2.11(2H, ddd, $J=11.5, 8.9, 5.6$ Hz, H-6), 2.07, 2.00(2H, m, H-15), 1.92, 1.41(2H, m, H-1), 1.88, 1.56(2H, m, H-12), 1.73(3H, s, H-27), 1.73, 1.39(2H, m, H-23), 1.69(1H, m, H-5), 1.65(2H, d, $J=5.1$ Hz, H-11), 1.62(1H, m, H-20), 1.59, 1.08(2H, m, H-22), 1.48(1H, m, H-17), 1.27(3H, s, H-30), 1.14(3H, s, H-28), 1.06(3H, s, H-29), 1.05(3H, s, H-21), 1.04(3H, d, $J=1.5$ Hz, H-19), 0.84(3H, d, $J=2.1$ Hz, H-18)。 $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ_{C} : 216.9(C-3), 145.1(C-8), 143.8(C-25), 118.2(C-7), 114.4(C-26), 89.8(C-24), 78.1(C-16), 62.5(C-17), 52.4(C-5), 49.9(C-14), 47.9(C-4), 47.9(C-9), 45.8(C-15), 45.4(C-13), 38.5(C-1), 35.1(C-10), 34.9(C-2), 34.1(C-20), 33.2(C-12), 30.7(C-22), 27.9(C-30), 27.8(C-23), 24.5(C-28), 24.4(C-6), 23.5(C-18), 21.6(C-29), 18.5(C-21), 18.2(C-11), 17.2(C-27), 12.8(C-19)。与文献[9]比对, 鉴定化合物8为 meliasenin L。

化合物9: 无色晶体, 熔点 $78\text{ }^\circ\text{C} \sim 82\text{ }^\circ\text{C}$, 分子式 $\text{C}_{30}\text{H}_{48}\text{O}_4$, ESI-MS m/z : 479 [M+Na] $^+$ 。 $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 5.29(1H, s, H-7), 4.93(2H, dt, $J=1.8, 0.9$ Hz, H-26), 4.85(1H, t, $J=1.7$ Hz, H-16), 4.03(1H, d, $J=5.3$ Hz, H-24), 2.75, 2.24(2H, m, H-2), 2.25(1H, m, H-9), 2.11, 1.48(2H, m, H-15), 2.10(2H, m, H-6), 1.99, 1.44(2H, m, H-1), 1.90, 1.60(2H, m, H-12), 1.73(3H, m, H-27), 1.71(1H, d, $J=3.4$ Hz, H-5), 1.69, 1.45(2H, m, H-23), 1.65, 0.98(2H, m, H-22), 1.62(1H, m, H-20), 1.57(2H, m, H-11), 1.44(1H, m, H-17), 1.25(3H, s, H-30), 1.11(3H, s, H-29), 1.05(3H, d, $J=1.6$ Hz, H-21), 1.04(3H, s, H-28), 1.01(3H, s, H-19), 0.82(3H, s, H-18)。 $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ_{C} : 216.8(C-3), 147.7(C-25), 145.1(C-8), 118.1(C-7), 111.1(C-26), 78.0(C-16), 76.5(C-24), 62.5(C-17), 52.4(C-5), 49.9(C-14), 47.9(C-4), 47.9(C-9), 45.7(C-15), 45.4(C-13), 38.4(C-1), 35.0(C-10), 34.9(C-2), 34.2(C-20), 33.2(C-12), 31.8(C-23), 30.8(C-22), 27.8(C-30), 24.5(C-28), 24.3(C-6), 23.5(C-18), 21.6(C-29), 18.6(C-21), 18.2(C-11), 17.4(C-27), 12.8(C-19)。与文献[8]比对, 鉴定化合物9为 meliastatin 5。

化合物10: 白色粉末, 分子式 $\text{C}_{30}\text{H}_{48}\text{O}_5$, ESI-MS m/z : 511 [M+Na] $^+$ 。 $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 5.32(1H, m, H-7), 4.17(1H, dd, $J=12.0, 3.5$ Hz, H-24), 4.02(1H, dd, $J=8.5, 4.9$ Hz, H-16), 3.50(1H, dd, $J=6.8, 4.0$ Hz, H-3), 2.66(1H, td, $J=11.2, 7.9$ Hz, H-20), 2.46, 1.97(2H, m, H-2), 2.30(1H, m, H-9), 2.09(1H, m, H-17), 1.91(2H, m, H-6), 1.83(2H, m, H-12), 1.90, 1.39(2H, m, H-22), 1.79(2H, dd, $J=12.1, 5.7$ Hz, H-23), 1.75(1H, d, $J=1.0$ Hz, H-5), 1.73(2H, d, $J=1.0$ Hz, H-15), 1.64, 1.56(2H, m, H-11), 1.53(2H, m, H-1), 1.33(3H, s, H-26), 1.31(3H, s, H-30), 1.26(3H, s, H-27), 0.96(3H, s, H-18), 0.94(3H, s, H-28), 0.93(3H, s, H-29), 0.81(3H, s, H-19)。 $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ_{C} : 178.5(C-21), 144.6(C-8), 118.8(C-7), 83.4(C-24), 77.6(C-16), 76.3(C-3), 71.1(C-25), 57.9(C-17), 50.0(C-14), 48.1(C-9), 45.8(C-13), 44.6(C-15), 43.9(C-5), 41.9(C-20), 37.4(C-4), 34.8(C-10), 33.6(C-12), 31.2(C-1), 27.8(C-28), 27.3(C-30), 26.2(C-26), 25.4(C-2), 24.4(C-27), 23.9(C-23), 23.2(C-18), 22.7(C-22), 21.8(C-29), 21.3(C-6), 17.9(C-11), 13.0(C-19)。与文献[10]比对, 鉴定化合物10为 toosendine G。

以上10种化合物均为甘遂烷型三萜类化合物, 其中化合物2和5首次从川楝中分离得到。

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