

玛咖化学成分的研究

梁文娟, 许洪波, 杨彩艳, 耿长安, 张雪梅, 陈纪军*

(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, 云南 昆明 650204)

[摘要] 对十字花科 Brassicaceae 植物马卡独行菜 *Lepidium meyenii* 的干燥根茎玛咖进行化学成分研究, 马卡独行菜的干燥根茎用 70% 提取, 依次用石油醚、乙酸乙酯、正丁醇萃取, 对各萃取部分采用各种柱色谱进行分离纯化, 通过波谱数据分析 ($^1\text{H-NMR}$, $^{13}\text{C-NMR}$, HRESIMS) 进行结构鉴定。从各萃取部分共分离鉴定出 18 个化合物, 包括 7 个生物碱, 4 个脂肪酸类化合物以及 7 个其他类化合物; 分别鉴定为 3-羟苄基氨基甲酸 [(3-hydroxybenzyl) carbamic acid (1)], phenylmethanamine (2), *N*-苄基甲醛 (*N*-benzylformamide (3)), *N*-苄基甲酰胺 (*N*-benzylacetamide (4)), pyridin-4-ylmethanamine (5), *n*-(4-methoxybenzyl) aniline (6), 尿嘧啶 (uracil (7)), 琥珀酸 (succinic acid (8)), 癸二酸 (decanedioic acid (9)), *n*-hexadecanoic acid methyl ester (10), 庚酸 (heptanoic acid (11)), solerole (12), 糠酸甲酯 (pyromucic acid methyl ester (13)), 5-羟甲基-2-呋喃羧醛 (14), 5-(甲氧基甲基)-1*H*-吡咯-2-羧醛 (15), 1,7-二羟基-2,3,4-三甲氧基呋喃酮 (1,7-dihydroxy-2,3,4-trimethoxyxanthone (16)), 1,7-二羟基-3,4-二甲氧基呋喃酮 (1,7-dihydroxy-3,4-dimethoxy-xanthone (17)), (+)-松脂醇 [(+)-pinoresinol (18)]。其中化合物 1~18 均为首次从玛咖中分离得到。

[关键词] 玛咖; 生物碱; 脂肪酸

Chemical constituents of *Lepidium meyenii*

LIANG Wen-juan, XU Hong-bo, YANG Cai-yan, GENG Chang-an, ZHANG Xue-mei, CHEN Ji-jun*

(State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, Chinese Academy of Sciences, Kunming 650204, China)

[Abstract] To study the chemical constituents of *Lepidium meyenii*, the air-dried rhizome of *L. meyenii* was extracted with 70% EtOH. The extract was condensed to a small amount of volume and extracted with petroleum ether, EtOAc and *n*-BuOH, successively. The compounds were isolated and purified by column chromatography, and identified based on spectral analyses ($^1\text{H-NMR}$, $^{13}\text{C-NMR}$, HRESIMS). Eighteen compounds were isolated from *L. meyenii*, including 7 alkaloids and 4 fatty acids and 7 other compounds. They were characterized as (3-hydroxybenzyl) carbamic acid (1), phenylmethanamine (2), *N*-benzylformamide (3), *N*-benzylacetamide (4), pyridin-4-ylmethanamine (5), *n*-(4-methoxybenzyl) aniline (6), uracil (7), succinic acid (8), decanedioic acid (9), *n*-hexadecanoic acid methyl ester (10), heptanoic acid (11), solerole (12), pyromucic acid methyl ester (13), 5-hydroxymethyl-2-furancarboxaldehyde (14), 5-(methoxymethyl)-1*H*-pyrrole-2-carbaldehyde (15), 1,7-dihydroxy-2,3,4-trimethoxyxanthone (16), 1,7-dihydroxy-3,4-dimethoxy-xanthone (17), (+)-pinoresinol (18). Meanwhile, compounds 1-18 were obtained from *L. meyenii* for the first time.

[Key words] *Lepidium meyenii*; alkaloids; fatty acids

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玛咖 (Maca) 是十字花科植物 Brassicaceae 马卡独行菜 *Lepidium meyenii* Walp. 的根茎, 原产于秘鲁,

目前云南等地多有栽培; 在秘鲁当地用来治疗妇科病、风湿病、呼吸道疾病等^[1]。玛咖具有消除焦虑、

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[通信作者] * 陈纪军, Tel/Fax: (0871) 65223265, E-mail: chenjj@mail.kib.ac.cn

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改善睡眠障碍的功能,对患有性功能障碍患者(选择性5羟色胺诱导,SSRI)摄取一定剂量玛咖(3 g·d⁻¹,连续12 d)能改善性功能障碍所引起的抑郁症状,效果与服用氟西汀,帕罗西汀,度洛西汀或氟伏沙明相当^[2];玛咖还具有提高记忆力,改善性功能,提高生殖力,调节新陈代谢,抗肿瘤^[1,3-6]等药理作用。文献报道玛咖中含有玛咖酰胺、玛咖烯、生物碱、芥子油苷、脂肪酸、甾醇等成分^[1]。为了寻找其活性成分,本实验从玛咖提取物的各萃取部分分离鉴定了18个化合物,包括7个生物碱,4个脂肪酸类化合物以及7个其他类化合物。其中化合物1~18均为首次从玛咖根茎中分离得到。

1 材料

质谱采用岛津LC-MS-IT-TOF(Shimadzu, Kyoto, Japan)测定[离子阱飞行时间质谱仪和高效液相色谱(Waters AutoSpec Premier P776 Waters, USA)];核磁共振谱采用Bruker AM-400, DRX-500, AVANCE III-600(Bruker, Bremerhaven, Germany)测定;熔点采用显微熔点仪SGW[®]X-4B测定(上海精密科学仪器有限公司,上海,中国)。薄层色谱硅胶、柱层析硅胶(200~300目)购自青岛美高集团有限公司;柱层析葡聚糖凝胶LH-20购自Pharmacia公司。分析纯甲醇购自天津化学试剂有限公司,纯净水购自娃哈哈集团有限公司;显色剂为H₂SO₄(10%)的乙醇溶液。样品于2013年6月采自云南丽江,由中国科学院昆明植物研究所雷立功研究员鉴定为玛咖*L. meyenii*。标本存放于中国科学院昆明植物研究所抗病毒与天然药物化学研究组(No. 20130626)。

2 提取与分离

玛咖干燥样品4.8 kg,经粉碎后用70%乙醇(12 L),回流提取3次,每次1 h,提取液经减压浓缩至1.2 L,分别用石油醚、乙酸乙酯和正丁醇萃取,回收溶剂得到石油醚部分(Fr. 1, 60 g),乙酸乙酯部分(Fr. 2, 132 g),正丁醇部分(Fr. 3, 1.8 kg)。

乙酸乙酯部位(Fr. 2, 132 g)以水(1 320.0 g)溶解,用乙醇-水(60:40)提取,得上清液(58.5 g)和沉淀(62.0 g)。上清液(58.5 g)经中压RP-18(Merck, 330 g, 8.0 cm×50.0 cm, 75~150 μm)用甲醇-水(10:90, 20:80, 30:70, 50:50, 100:0)洗脱,得5个流分:Fr. 2-1~Fr. 2-5。Fr. 2-4(20.0 g)吸附于40.0 g硅胶(200~300目),室温挥干,通过硅胶柱色谱(230.0 g, 7.0 cm×10.0 cm),水-甲醇-二乙

胺-氯仿体系(1:10:1:90, 2:20:1:80, 3:30:1:70, 5:50:1:50)梯度洗脱,再经Sephadex LH-20(30 g, 1.4 cm×120.0 cm, 甲醇)多次纯化,得化合物3-羟苄基氨基甲酸[(3-hydroxybenzyl) carbamic acid, **1**](10 mg),癸二酸(**9**, 5 mg),庚酸(**11**, 11 mg)。Fr. 2-2(10.0 g)吸附于20.0 g硅胶(200~300目),室温下挥干溶剂,通过硅胶柱色谱(120.0 g, 7.0 cm×10.0 cm)经甲醇-氯仿体系梯度洗脱(10:90, 20:80, 30:70, 50:50, 100:0),再经Sephadex LH-20(30 g, 1.4 cm×120.0 cm, 甲醇)多次纯化,得化合物phenylmethanamine(**2**, 5 mg),pyridin-4-ylmethanamine(**5**, 5 mg),*n*-(4-methoxybenzyl) aniline(**6**, 5 mg)。

玛咖正丁醇部分(Fr. 3, 1.8 kg)用70%乙醇沉淀,取上清液,回收溶剂得1.0 kg,通过硅胶柱色谱(5 kg, 25×135 cm),水-甲醇-氯仿(1:10:90, 2:20:80, 3:30:70, 4:40:60, 5:50:50)体系梯度洗脱,得到5个流分Fr. 3-1~Fr. 3-5。Fr. 3-4(18.0 g)吸附于40.0 g硅胶(200~300目),室温挥干溶剂,通过硅胶柱色谱(230 g, 7.0 cm×10.0 cm),水-甲醇-二乙胺-氯仿体系(1:10:1:90, 2:20:1:80, 3:30:1:70, 5:50:1:50)梯度洗脱,再经Sephadex LH-20(30 g, 1.4 cm×120.0 cm, 甲醇)多次纯化,得化合物尿嘧啶(**7**, 20 mg),琥珀酸(**8**, 12 mg),*n*-hexadecanoic acid methyl ester(**10**, 10 mg),solerole(**12**, 36 mg),糠酸甲酯(**13**, 25 mg),5-(methoxymethyl)-1H-pyrrole-2-carbaldehyde(**15**, 15 mg)。

石油醚部分(Fr. 1, 60 g)吸附于等量硅胶,室温挥干溶剂,经硅胶柱(8.0 cm×50 cm, 600 g)色谱,用氯仿-甲醇(100:0, 10:90, 20:80, 30:70, 50:50, 100:0)梯度洗脱,得到Fr. 1-1~Fr. 1-6共6个组分。组分Fr. 1-1(31.5 g)通过硅胶柱色谱(400 g, 7.0 cm×50.0 cm),乙酯-石油醚(5:95, 10:90, 15:85)洗脱,再经Sephadex LH-20(30 g, 1.4×120.0 cm, 甲醇)多次纯化,得化合物*N*-苄基甲醛(**3**, 12 mg),*N*-苄基乙酰胺(**4**, 14 mg),5-hydroxymethyl-2-furancarboxaldehyde(**14**, 8 mg),1,7-dihydroxy-2,3,4-trimethoxyxanthone(**16**, 6 mg),1,7-dihydroxy-3,4-dimethoxy-xanthone(**17**, 2 mg)和(+)-松脂醇(**18**, 81 mg)。

3 结构鉴定

化合物**1** 白色粉末, C₈H₉NO₃, ESI-MS *m/z* 202 [M+Cl]⁻; ¹H-NMR(CD₃OD, 600 MHz) δ: 7.11

(1H, m, H-8) δ : 7.75 (2H, overlapped, H-5, H-9) δ : 6.67 (1H, d, $J = 8.4, 1.8$ Hz, H-7) δ : 4.21 (2H, s, H-3); $^{13}\text{C-NMR}$ (CD₃OD, 150 MHz) δ : 177.2 (C-1), 43.6 (C-3), 138.4 (C-4), 115.0 (C-5), 158.8 (C-6), 117.1 (C-7), 130.7 (C-8), 121.4 (C-9); 以上数据与文献[7]对照, 鉴定为 3-羟苄基氨基甲酸 [(3-hydroxybenzyl) carbamic acid]。

化合物 2 白色粉末, C₇H₉N, ESI-MS m/z 142 [M + Cl]⁻。 $^1\text{H-NMR}$ (CD₃OD, 600 MHz) δ : 7.45 (2H, overlapped, H-5, 7) δ : 7.40 (3H, overlapped, H-4, 6, 8) δ : 4.13 (2H, s, H-2); $^{13}\text{C-NMR}$ (CD₃OD, 150 MHz) δ : 44.5 (C-2), 134.7 (C-3), 130.2 (C-4, 8), 130.3 (C-5, 7), 130.1 (C-6); 以上数据与文献[8]对照, 鉴定为 phenylmethanamine。

化合物 3 白色粉末, C₈H₉NO₅, ESI-MS m/z 136 [M + H]⁺。 $^1\text{H-NMR}$ (CD₃OD, 400 MHz) δ : 8.14 (1H, br s, H-1) δ : 7.34 (5H, overlapped, H-4, 5, 6, 7, 8) δ : 4.40 (2H, s, H-2); $^{13}\text{C-NMR}$ (CD₃OD, 100 MHz) δ : 163.6 (C-1), 42.7 (C-2), 139.4 (C-3), 129.6 (C-4, 8), 128.6 (C-5, 7), 128.4 (C-6); 以上数据与文献[9]对照, 鉴定为 *N*-苄基甲酰胺 (*N*-benzylformamide)。

化合物 4 白色粉末, C₉H₁₁NO, ESI-MS m/z 150 [M + H]⁺。 $^1\text{H-NMR}$ (CD₃OD, 400 MHz) δ : 7.28 (5H, overlapped, H-3, 4, 5, 6, 7) δ : 4.39 (2H, s, H-1), 1.98 (3H, s, COCH₃); $^{13}\text{C-NMR}$ (CD₃OD, 100 MHz) δ : 42.7 (C-1), 139.9 (C-2), 129.5 (C-3, 7), 128.6 (C-4, 6), 128.2 (C-5), 173.1 (COCH₃), 23.1 (COCH₃); 以上数据与文献[10]对照, 鉴定为 *N*-苄基甲酰胺 (*N*-benzylacetamide)。

化合物 5 白色粉末, C₇H₉N, ESI-MS m/z 142 [M + Cl]⁻。 $^1\text{H-NMR}$ (CD₃OD, 600 MHz) δ : 7.47 ~ 7.42 (4H, overlapped, H-4, 5, 7, 8) δ : 4.12 (2H, s, H-2); $^{13}\text{C-NMR}$ (CD₃OD, 150 MHz) δ : 44.5 (C-2), 144.6 (C-3), 130.2 (C-4, 8), 150.4 (C-5, 7); 以上数据与文献[11]对照, 鉴定为 pyridin-4-ylmethanamine。

化合物 6 无色油状, C₁₄H₁₅NO, ESI-MS m/z 248 [M + Cl]⁻。 $^1\text{H-NMR}$ (CD₃OD, 600 MHz) δ : 7.50 (4H, overlapped, H-4, 8, 3', 5') δ : 7.40 (4H, overlapped, H-5, 7, 2', 6') δ : 6.78 (1H, d, H-4') δ : 4.10 (2H, s, H-2) δ : 3.88 (3H, s, H-9); $^{13}\text{C-NMR}$ (CD₃OD, 150 MHz) δ : 44.5 (C-2), 134.9 (C-3), 130.4 (C-4, 8),

115.6 (C-5, 7), 151.2 (C-6), 56.4 (C-9), 148.4 (C-1'), 114.0 (C-2', 6'), 129.8 (C-3', 5'), 124.8 (C-4'); 以上数据与文献[12]对照, 鉴定为 *N*-(4-methoxybenzyl) aniline。

化合物 7 白色粉末, mp 338 °C, C₄H₄N₂O₂, ESI-MS m/z 113 [M + H]⁺。 $^1\text{H-NMR}$ (DMSO-*d*₆, 400 MHz) δ : 7.37 (1H, d, $J = 7.5$ Hz, H-6) δ : 5.44 (1H, d, $J = 7.5$ Hz, H-5)。 $^{13}\text{C-NMR}$ (DMSO-*d*₆, 100 MHz) δ : 166.2 (C-4), 153.4 (C-2), 102.1 (C-5), 144.1 (C-6); 以上数据与文献[13]对照, 鉴定为 尿嘧啶 (uracil)。

化合物 8 无色油状, C₄H₆O₄, ESI-MS m/z 117 [M - H]⁻。 $^1\text{H-NMR}$ (CDCl₃, 400 MHz) δ : 2.56 (4H, m, H-2, 3)。 $^{13}\text{C-NMR}$ (CDCl₃, 100 MHz) δ : 176.1 (C-1, 4), 29.8 (C-2, 3); 以上数据与文献[14]对照, 鉴定为 琥珀酸 (succinic acid)。

化合物 9 无色油状, C₁₀H₁₈O₄, ESI-MS m/z 225 [M + Na]⁺。 $^1\text{H-NMR}$ (CD₃OD, 400 MHz) δ : 3.21 (4H, overlapped, H-2, H-9) δ : 2.20 (4H, overlapped, H-5, 7) δ : 1.51 (4H, overlapped, H-3, 8) δ : 1.30 (4H, overlapped, H-4, 6); $^{13}\text{C-NMR}$ (CD₃OD, 100 MHz) δ : 175.7 (C-1, 10), 37.1 (C-2, 9), 26.7 (C-3, 8), 27.6 (C-4, 7), 30.2 (C-5, 6); 以上数据与文献[15]对照, 鉴定为 癸二酸 (decanedioic acid)。

化合物 10 无色油状, C₁₇H₃₄O₂, ESI-MS m/z 293 [M + Na]⁺。 $^1\text{H-NMR}$ (CDCl₃, 400 MHz) δ : 3.66 (3H, s, H-17) δ : 2.33 ~ 1.30 (3H, overlapped, H-2 ~ 15) δ : 0.86 (3H, t, $J = 6.8$ Hz, H-16)。 $^{13}\text{C-NMR}$ (CDCl₃, 100 MHz) δ : 174.4 (C-1), 22.6 ~ 34.0 (14 × C, C-2 ~ 15), 14.1 (C-16), 51.4 (C-17); 以上数据与文献[16]对照, 鉴定为 *n*-hexadecanoic acid methyl ester。

化合物 11 无色油状, C₇H₁₄O₂, ESI-MS m/z 130 [M + Na]⁺。 $^1\text{H-NMR}$ (CD₃OD, 400 MHz) δ : 3.31 (4H, overlapped, H-2, 3) δ : 2.94 (6H, overlapped, H-4, 5, 6) δ : 1.18 (3H, t, $J = 7.3$ Hz, H-7); $^{13}\text{C-NMR}$ (CD₃OD, 100 MHz) δ : 175.9 (C-1), 43.5 (C-2), 30.2 (C-3), 27.5 (C-4), 37.0 (C-5), 26.7 (C-6), 11.6 (C-7); 以上数据与文献[17]对照, 鉴定为 庚酸 (heptanoic acid)。

化合物 12 油状物, C₆H₁₀O₃, ESI-MS m/z 131

[M + H]⁺; ¹H-NMR (CDCl₃, 400 MHz) δ: 4.30 (1H, m, H-4), 3.76 (1H, m, H-5), 2.21 (2H, t, J = 4.8 Hz, H-2), 1.38 (2H, m, H-3), 1.13 (3H, d, J = 6.8 Hz, H-6)。¹³C-NMR (CDCl₃, 100 MHz) δ: 178.2 (C-1), 29.1 (C-2), 20.7 (C-3), 84.2 (C-4), 67.3 (C-5), 18.1 (C-6); 以上数据与文献 [18] 对照鉴定为 solerole。

化合物 13 C₆H₆O₃, ESI-MS *m/z* 127 [M + H]⁺。¹H-NMR (CD₃OD, 600 MHz) δ: 8.17 (1H, s, H-7), 8.03 (1H, d, J = 1.8 Hz, H-5), 7.92 (1H, dd, J = 3.6, 1.8 Hz, H-4), 7.29 (1H, d, J = 3.6 Hz, H-3), 3.92 (3H, s, OMe)。¹³C-NMR (CD₃OD, 150 MHz) δ: 159.2 (C-2), 128.3 (C-3), 123.9 (C-4), 139.3 (C-5), 166.6 (C-6), 53.0 (C-7); 以上数据与文献 [19] 对照鉴定为糠酸甲酯 (pyromucic acid methyl ester)。

化合物 14 C₆H₆O₃, 黄色粉末, mp 35 ~ 36 °C, ESI-MS *m/z* 127 [M + H]⁺。¹H-NMR (CDCl₃, 400 MHz) δ: 9.54 (1H, s, H-7), 7.20 (1H, d, J = 3.6 Hz, H-3), 6.50 (1H, d, J = 3.6 Hz, H-4), 3.22 (2H, s, H-6)。¹³C-NMR (CDCl₃, 100 MHz) δ: 160.8 (C-2), 152.2 (C-3), 110.0 (C-4), 123.1 (C-5), 57.4 (C-6), 177.7 (C-7); 以上数据与文献 [20] 对照鉴定为 5-hydroxymethyl-2-furancarboxaldehyde。

化合物 15 C₇H₉NO₂, ESI-MS *m/z* 140 [M + H]⁺。¹H-NMR (CDCl₃, 400 MHz) δ: 9.65 (1H, br s, H-1), 9.47 (1H, s, H-7), 3.37 (3H, s, OMe), 6.91 (1H, d, J = 2.6 Hz, H-3), 6.21 (1H, d, J = 2.6 Hz, H-4), 4.49 (2H, s, H-6)。¹³C-NMR (CDCl₃, 100 MHz) δ: 132.8 (C-2), 121.5 (C-3), 109.8 (C-4), 137.6 (C-5), 67.0 (C-6), 178.9 (C-7), 58.4 (OMe); 以上数据与文献 [21] 对照鉴定为 5-(methoxymethyl)-1H-pyrrole-2-carbaldehyde。

化合物 16 黄色粉末, mp 163 ~ 166 °C, C₁₆H₁₄O₇, ESI-MS *m/z* 317 [M - H]⁻; ¹H-NMR (DMSO, 400 MHz) δ: 7.37 (2H, overlapped, H-5, 8), 7.20 (1H, dd, J = 1.8, 6.7 Hz, H-6), 4.05 (3H, s, OMe), 3.84 (6H, overlapped, 2 × OMe); ¹³C-NMR (DMSO, 100 MHz) δ: 155.4 (C-1), 136.2 (C-2), 151.2 (C-3), 147.5 (C-4), 151.3 (C-4a), 153.3 (C-4b), 120.1 (C-5), 126.0 (C-6), 155.6 (C-7), 109.1 (C-8), 121.6 (C-8a), 105.7 (C-8b), 182.7 (C-9),

62.4, 62.2, 61.5 (2 × OMe); 以上数据与文献 [22] 对照鉴定为 1,7-二羟基-2,3,4-三甲氧基呋酮 (1,7-dihydroxy-2,3,4-trimethoxyxanthone)。

化合物 17 黄色粉末, mp 272 ~ 278 °C, C₁₅H₁₂O₆, ESI-MS *m/z* 289 [M + H]⁺; ¹H-NMR (DMSO-*d*₆, 600 MHz) δ: 7.56 (1H, d, J = 9.0 Hz, H-5), 7.42 (1H, d, J = 3.0 Hz, H-8), 7.31 (1H, dd, J = 9.0, 3.0 Hz, H-6), 6.58 (1H, s, H-2), 3.93 (3H, s, OMe), 3.78 (3H, s, OMe); ¹³C-NMR (DMSO-*d*₆, 100 MHz) δ: 148.9 (C-1), 94.8 (C-2), 158.1 (C-3), 128.1 (C-4), 149.2 (C-4a), 154.1 (C-4b), 119.4 (C-5), 124.9 (C-6), 159.7 (C-7), 108.03 (C-8), 119.4 (C-8a), 102.3 (C-8b), 180.4 (C-9), 61.0, 56.6 (2 × OMe); 以上数据与文献 [22] 对照鉴定为 1,7-二羟基-3,4-二甲氧基呋酮 (1,7-dihydroxy-3,4-dimethoxy-xanthone)。

化合物 18 白色粉末, C₂₀H₂₂O₆, ESI-MS *m/z* 381 [M + Na]⁺; ¹H-NMR (CD₃OD, 400 MHz) δ: 6.93 (2H, br s, H-2, 2'), 6.77 (4H, m, overlapped, H-5, 6, 5', 6'), 4.68 (2H, overlapped, H-7, 7'), 4.19 (2H, overlapped, H-9a, 9a'), 3.80 (8H, overlapped, H-9b, 9b', 10, 10b'), 3.10 (2H, overlapped, H-8, 8'); ¹³C-NMR (CD₃OD, 125 MHz) δ: 133.8 (C-1, 1'), 111.1 (C-2, 2'), 149.1 (C-3, 3'), 147.3 (C-4, 4'), 116.1 (C-5, 5'), 120.5 (C-6, 6'), 87.5 (C-7, 7'), 56.4 (C-8, 8'), 72.6 (C-9, 9'), 55.3 (C-10, 10'); 以上数据与文献 [23] 对照鉴定为 (+)-松脂醇 [(+)-pinosresinol]。

此外, 还从玛咖中分离得到 *N*-benzylhexadecanamide, 蔗糖, 葡萄糖, 对羟基苯甲酸等成分。

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