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望谟崖摩和曲枝崖摩的化学成分研究

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摘 要: 为了寻找生物活性成分, 采用色谱分离法对望谟崖摩和曲枝崖摩进行了化学成分研究。根据波谱学分析鉴定了化合物结构。从两种植物中分离得到了 15 个化合物, 分别为 8(17), 13(*E*)-半日花-二烯-15, 19-二酸(**1**), 8(17), 13(*E*)-半日花-二烯-19-酸甲酯-15-醛(**2**), 15-O-乙酰基-8(17), 13(*E*)-半日花-二烯-19-酸(**3**), 15-羟基-8(17), 13(*E*)-半日花-二烯-19-酸(**4**), 15, 19-二羟基-8(17), 13(*E*)-半日花-二烯(**5**), 19-羟基-8(17), 13(*E*)-半日花-二烯-15-醛(**6**), 19-羟基-8(17), 13(*Z*)-半日花-二烯-15-醛(**7**), 8(17), 13(*E*)-半日花-二烯-19-酸-15-醛(**8**), 8(17), 13(*Z*)-半日花-二烯-19-酸-15-醛(**9**), (+)-儿茶素(**10**), β-香树素(11), 豆甾-5-烯-3β, 7α-二醇(**12**), 东莨菪内酯(**13**), β-谷甾醇(**14**), 胡萝卜苷(**15**)。上述化合物均为首次从崖摩属植物中分离得到的半日花烷型二萜。

关键词: 望谟崖摩; 曲枝崖摩; 半日花烷二萜

中图分类号: Q946.91; R284.2 文献标识码: A

Chemical Constituents from *Amoora ouangtliensis* and *A. stellato-squamosa*

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Abstract The phytochemical investigations on *Amoora ouangtliensis* and *A. stellato-squamosa* were conducted in order to look for bioactive compounds. Nine diterpenoids were obtained by the method of column chromatography, and on the basis of spectral analysis they were identified as 8(17), 13(E)-labdadien-15, 19-dioic acid(**1**), methyl 8(17), 13(*E*)-labdadien-19-oic acid-15-oate(**2**), 15-acetoxy-8(17), 13(*E*)-labdadien-19-oic acid(**3**), 15-hydroxy-8(17), 13(*E*)-labdadien-19-oic acid (4), 8(17), 13(*E*)-labdadien-15, 19-diol(**5**), 19-hydroxy-8(17), 13(*E*)-labdadien-15-al(**6**), 19-hydroxy-8(17), 13(*Z*)-labdadien-15-al(7), 8(17), 13(*E*)-labdadien-19-oic acid-15-al(**8**), 8(17), 13(*Z*)-labdadien-19-oic acid-15-al(**9**), and six other compounds, (+)-catechin(**10**), β-amyrin(**11**), stigmast-5-en-3β, 7α-diol(**12**), scopoletin(**13**), β-sitosterol(**14**), daucosterol(**15**). This is the first report of labdane-type diterpene from the genus *Amoora*.

Key words *Amoora ouangtliensis*; *A. stellato-squamosa*; labdane-type diterpene

Introduction

The genus *Amoora* (Meliaceae), mainly distributed in India and Malaysia, comprises ca 25-30 species, of which six have been found in Yunnan Province, China[1]. Previously, we reported neoclerodane, triterpenes from this genus[2-5]. In this paper, we continued to describe the isolation and identification of nine labdane-type diterpenes from *Amoora ouangtliensis* and *A. stellato-squamosa*, on the basis of spectral evidences, they were identified as 8(17), 13(*E*)-labdadien-15, 19-dioic acid(**1**), methyl 8(17), 13(*E*)-labdadien-19-oic acid-15-oate(2), 15-acetoxy-8(17), 13(*E*)-labdadien-19-oic acid(**3**), 15-hydroxy-8(17), 13(*E*)-labdadien-19-oic acid(**4**), 8(17), 13(*E*)-labdadien-15, 19-diol(**5**), 19-hydroxy-8(17), 13(*E*)-labdadien-15-al

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(**6**), 19-hydroxy-8(17), 13(*Z*)-labdadien-15-al(**7**), 8(17), 13(*E*)-labdadien-19-oic acid-15-al(**8**) and 8(17), 13(*Z*)-labdadien-19-oic acid-15-al(**9**). This is the first report of labdane-type diterpene from the genus *Amoora*. Besides, six other compounds, (+)-catechin (**10**), β-amyrin(**11**), stigmast-5-en-3β, 7α-diol(**12**), scopoletin(**13**), β-sitosterol(**14**), daucosterol(**15**), were obtained from the same source.

Experimental

Apparatus and materials

MS spectra were obtained with a VG Auto Spec-3000 spectrometer, at 70 eV for EI. NMR Spectra were recorded on a Bruker DRX-500 spectrometers with TMS as internal standard, δ in ppm, *J* in Hz. Silica gel(200-300 mesh) for CC and GF_{254} for analytical TLC were from the Qingdao Marine Chemical Factory, P. R. China.

The barks of *A. ouengliensis* and the twigs of *A. stellato-squamosa* were collected in Xishuangbanna of Yunnan Province, P. R. China, in January 2002. The plant was identified by Prof. Jing-Yun Cui, Xishuangbanna Tropical Botanical Garden, Academia Sinica, China.

Extraction and isolation

The air-dried twigs of *A. stellato-squamosa* (9.0 kg) were extracted with EtOH/H_2O 9∶1 at room temperature for 3 times. After evaporation, the residue was suspended in H_2O and extracted with EtOAc. The EtOAc extract(110 g) was subjected to CC(SiO_2; petroleum ether/AcOEt 1∶0 to 8∶2) to afford nine fractions(Fr. 1-9), as judged by TLC. Fr. 2(50 g) and Fr. 3(27 g) were repeatedly chromatographed on SiO_2(petroleum ether/AcOEt 49∶1 to 1∶1, 10∶1 to 1∶1, respectively), affording **2**(8 mg), **14**(590 mg) from Fr. 2; **3**(7 mg), **4**(11 mg), **13**(410 mg) from Fr. 3. Fr. 4(10 g) and Fr. 5(9 g) were repeatedly chromatographed(1. SiO_2, petroleum ether/AcOEt 9∶1 to 1∶1; 2. RP-18 gel, MeOH/H_2O 1∶1 to 1∶0 respectively), affording **5**(114 mg), **10**(17 mg) from Fr. 4; **1**(9 mg) from Fr. 5. Fr. 9(21 g) was repeatedly subjected to CC over SiO_2 ($CHCl_3$/MeOH 9∶1 to 7∶3) to give **15**(40 mg). The air-dried barks of *A. ouengliensis* (7.0 kg) were extracted with EtOH/H_2O 9∶1 at refluxed temperature for 3 times(4 h for each time). After evaporation, the residue was suspended in H_2O and extracted with $CHCl_3$. The $CHCl_3$ extract(110 g) was subjected to CC(SiO_2; petroleum ether/AcOEt 1∶0 to 8∶2) to afford eight fractions (Fr. 1-8), as judged by TLC. Fr. 3(9 g) was repeatedly chromatographed on(1. SiO_2, petroleum ether/Me_2CO 8∶2 to 1∶1; 2. RP-18 gel, MeOH/H_2O 1∶1 to 1∶0, successively), affording **6** and **7**(93 mg), **8** and **9**(61 mg), **12**(32 mg). Fr. 4(20 g) and Fr. 5(9.5 g) were repeatedly chromatographed on (1. SiO_2, $CHCl_3$/Me_2CO 9∶1; 2. RP-18 gel, MeOH/H_2O 1∶1 to 1∶0 successively), yielding **9**(9 mg) from Fr. 4 and **11**(29 mg). Fr. 6(5.5 g) and Fr. 7(12 g) were repeatedly subjected to CC over SiO_2 ($CHCl_3$/Me_2CO, 4∶1 and 3∶1 respectively), producing **11**(110 mg) from Fr. 6, **12**(57 mg) from Fr. 7. Fr. 8(27 g) was repeatedly subjected to CC over SiO_2($CHCl_3$/MeOH 9∶1) to give **15** (230 mg).

Identification

8(17), 13(*E*)-Labdadien-15, 19-dioic acid(1) White crystal, ^{1}H NMR($CDCl_3$, 400 MHz) δ: 5.73 (1H, *brs*, H-14), 2.20(3H, d, *J* = 1.0 Hz, Me-16), 0.63(3H, s, Me-20); ^{13}C NMR($CDCl_3$, 100 MHz) δ: 39.9(t, C-1), 20.8(t, C-2), 39.5(t, C-3), 44.6(s, C-4), 56.8(d, C-5), 27.1(t, C-6), 39.0(t, C-7), 149.0(s, C-8), 56.3(d, C-9), 41.1(s, C-10), 22.5 (t, C-11), 40.4(t, C-12), 161.1(s, C-13), 116.2(d, C-14), 176.7(s, C-15), 18.7(q, C-16), 106.7(t, C-17), 29.3(q, C-18), 176.8(s, C-19), 13.3(q, C-20). The data were in accordance with those reported[6].

Methyl 8(17), 13(*E*)-labdadien-19-oic acid-15-oate(2) White crystal, EI-MS *m/z*: 348[M]$^+$(3), 333(7), 302(8), 274(13), 235(15), 189(26), 121 (60), 114(44), 95(50), 82(100), 69(34), 55 (60). ^{1}H NMR($CDCl_3$, 400 MHz) δ: 5.62(1H, s, H-14), 4.85(1H, s, H-17a), 4.48(1H, s, H-17b), 3.66 (3H, s, OCH_3), 2.13(3H, s, Me-16), 1.22(3H, s, Me-18), 0.58(3H, s, Me-20); ^{13}C NMR($CDCl_3$, 100 MHz) δ: 39.0(t, C-1), 19.8(t, C-2), 37.9(t, C-3), 44.4(s, C-4), 56.2(d, C-5), 26.0(t, C-6), 38.6(t, C-7), 147.6(s, C-8), 55.3(d, C-9), 40.4(s, C-10),

21.6(t, C-11), 39.7(t, C-12), 161.0(s, C-13), 115.0(d, C-14), 167.3(s, C-15), 18.9(q, C-16), 106.6(t, C-17), 29.0(q, C-18), 183.4(s, C-19), 12.8(q, C-20), 50.8(q, 15-OCH_3). The data were identical to those of literature[6].

15-Acetoxy-8(17), 13(*E*)-labdadien-19-oic acid (3) Colorless oily solid, ^{1}H NMR($CDCl_3$, 400MHz) δ 5.39(1H, d, *J* = 7.0Hz, H-14), 4.88(1H, s, H-17a), 4.63(1H, s, H-17b), 4.75(2H, d, *J* = 7.0Hz, H-15), 1.71(3H, s, Me-16), 1.29(3H, s, Me-18), 2.01(3H, s, CH_3CO); ^{13}C NMR($CDCl_3$, 100MHz) δ 39.0(t, C-1), 19.8(t, C-2), 37.9(t, C-3), 44.1(s, C-4), 55.3(d, C-5), 26.0(t, C-6), 38.3(t, C-7), 147.9(s, C-8), 56.3(d, C-9), 40.3(s, C-10), 21.7(t, C-11), 38.6(t, C-12), 142.7(s, C-13), 118.0(d, C-14), 61.3(t, C-15), 16.4(q, C-16), 106.3(t, C-17), 28.9(q, C-18), 183.5(s, C-19), 12.7(q, C-20), 20.9(q, 15-CH_3CO), 171.1(s, 15-$COCH_3$). These data were consistent with those reported[7].

15-Hydroxy-8(17), 13(*E*)-labdadien-19-oic acid (4) Colorless oily solid, EI-MS *m/z*: 320[M]$^+$(3), 305(24), 302(25), 287(28), 274(20), 259(24), 241(24), 235(19), 189(70), 161(29), 147(40), 133(52), 121(100), 107(49), 93(42), 81(47), 67(18), 55(12). ^{1}H NMR($CDCl_3$, 400MHz) δ 5.35(1H, d, *J* = 6.7Hz, H-14), 4.83(1H, s, H-17a), 4.50(1H, s, H-17b), 4.13(2H, d, *J* = 6.7Hz, H-15), 1.64(3H, s, Me-16), 1.20(3H, s, Me-18), 0.57(3H, s, Me-20); ^{13}C NMR($CDCl_3$, 100MHz) δ 39.1(t, C-1), 19.9(t, C-2), 37.9(t, C-3), 44.2(s, C-4), 56.3(d, C-5), 26.0(t, C-6), 38.7(t, C-7), 147.9(s, C-8), 55.5(d, C-9), 40.4(s, C-10), 21.9(t, C-11), 38.4(t, C-12), 140.5(s, C-13), 122.9(d, C-14), 59.4(t, C-15), 16.3(q, C-16), 106.5(t, C-17), 29.0(q, C-18), 183.7(s, C-19), 12.8(q, C-20). The data were identical to those reported[8].

8(17), 13(*E*)-Labdadien-15, 19-diol(5) EI-MS *m/z*: 306[M]$^+$(4), 291(12), 275(45), 257(73), 189(45), 161(64), 153(90), 135(82), 109(82), 107(100), 95(85), 81(74), 79(48), 67(38). ^{1}H NMR($CDCl_3$, 400MHz) δ 5.36(1H, t, *J* = 6.9Hz, H-14), 4.80(1H, s, H-17a), 4.49(1H, s, 17b), 4.13(2H, d, *J* = 6.9Hz), 3.73, 3.36(each 1H, d, *J* = 11.0Hz, H-19), 1.65(3H, s, Me-16), 0.95(3H, s, Me-18), 0.63(3H, s, Me-20); ^{13}C NMR($CDCl_3$, 100MHz) δ 38.3(t, C-1), 19.0(t, C-2), 35.4(t, C-3), 39.5(s, C-4), 56.2(d, C-5), 24.4(t, C-6), 38.6(t, C-7), 148.0(s, C-8), 56.3(d, C-9), 38.8(s, C-10), 22.2(t, C-11), 39.0(t, C-12), 140.5(s, C-13), 123.0(d, C-14), 59.4(t, C-15), 16.3(q, C-16), 106.6(t, C-17), 27.0(q, C-18), 65.0(t, C-19), 15.3(q, C-20). The data were equal to those of literature[6].

19-Hydroxy-8(17), 13(*E*)-labdadien-15-al(6) Colorless crystal, ^{1}H NMR($CDCl_3$, 400MHz) δ 9.93(1H, d, *J* = 8.1Hz, H-15), 5.82(1H, d, *J* = 7.8Hz, H-14), 4.80(1H, s, H-17a), 4.44(1H, s, H-17b), 3.70, 3.34(1H, d, *J* = 10.9Hz, H-19), 2.11(3H, s, Me-16), 0.93(3H, s, Me-18), 0.62(3H, s, Me-20); ^{13}C NMR($CDCl_3$, 100MHz) δ 38.4(t, C-1), 18.9(t, C-2), 35.2(t, C-3), 39.4(s, C-4), 56.2(d, C-5), 24.4(t, C-6), 38.4(t, C-7), 147.5(s, C-8), 56.2(d, C-9), 38.8(s, C-10), 22.3(t, C-11), 39.0(t, C-12), 164.9(s, C-13), 127.1(d, C-14), 191.4(d, C-15), 17.6(q, C-16), 106.6(t, C-17), 27.0(q, C-18), 64.8(t, C-19), 15.2(q, C-20). The data were identical to those of literature[9].

19-Hydroxy-8(17), 13(*Z*)-labdadien-15-al(7) Colorless crystal, ^{1}H NMR($CDCl_3$, 400MHz) δ 9.78(1H, d, *J* = 8.1Hz, H-15), 5.84(1H, d, *J* = 7.8Hz, H-14), 4.84(1H, s, H-17a), 4.50(1H, s, H-17b), 3.70, 3.34(1H, d, *J* = 10.9Hz, H-19), 1.93(3H, s, Me-16), 0.93(3H, s, Me-18), 0.62(3H, s, Me-20); ^{13}C NMR($CDCl_3$, 100MHz) δ 38.4(t, C-1), 18.9(t, C-2), 35.2(t, C-3), 39.4(s, C-4), 56.2(d, C-5), 24.4(t, C-6), 38.4(t, C-7), 147.5(s, C-8), 56.2(d, C-9), 38.8(s, C-10), 21.3(t, C-11), 31.0(t, C-12), 164.8(s, C-13), 129.0(d, C-14), 191.1(d, C-15), 24.5(q, C-16), 106.8(t, C-17), 27.0(q, C-18), 64.8(t, C-19), 15.2(q, C-20). The data were in accordance with those reported[9].

8(17), 13(*E*)-Labdadien-19-oic acid-15-al(8) Colorless oily solid, ^{1}H NMR($CDCl_3$, 400MHz) δ 9.98(1H, d, *J* = 8Hz, H-15), 5.86(1H, d, *J* = 8Hz, H-14), 4.86, 4.47(each 1H, s, H-17), 2.14(3H, s, Me-16), 1.22(3H, s, Me-18), 0.58(3H, s, Me-20);

^{13}C NMR (CDCl$_3$, 100 MHz) δ 39.1 (t, C-1), 19.9 (t, C-2), 37.9 (t, C-3), 44.1 (s, C-4), 56.1 (d, C-5), 26.0 (t, C-6), 38.6 (t, C-7), 147.5 (s, C-8), 56.4 (d, C-9), 40.5 (s, C-10), 21.4 (t, C-11), 39.5 (t, C-12), 164.8 (s, C-13), 127.2 (d, C-14), 191.4 (d, C-15), 17.7 (q, C-16), 106.6 (t, C-17), 28.9 (q, C-18), 182.8 (s, C-19), 12.8 (q, C-20). These data were consistent with those reported[7].

8(17), 13(*Z*)-Labdadien-19-oic acid-15-al (9) Colorless oily solid, ^{1}H NMR (CDCl$_3$, 400 MHz) δ 9.82 (1H, d, *J* = 8.1 Hz, H-15), 5.89 (1H, d, *J* = 8.0 Hz, H-14), 4.92, 4.56 (1H, s, H-17), 1.97 (3H, s, Me-16), 1.24 (3H, s, Me-18), 0.61 (3H, s, Me-20); ^{13}C NMR (CDCl$_3$, 100 MHz) δ 39.1 (t, C-1), 19.8 (t, C-2), 37.8 (t, C-3), 44.2 (s, C-4), 55.4 (d, C-5), 26.0 (t, C-6), 38.6 (t, C-7), 147.5 (s, C-8), 56.2 (d, C-9), 40.5 (s, C-10), 21.4 (t, C-11), 39.5 (t, C-12), 164.8 (s, C-13), 127.2 (d, C-14), 191.4 (d, C-15), 24.3 (q, C-16), 106.6 (t, C-17), 29.0 (q, C-18), 183.7 (s, C-19), 12.8 (q, C-20). The data were in accordance with those reported[7].

(+)-Catechin (10) Pale yellow crystal, EI-MS *m/z* 290 [M]$^+$ (52), 279 (2), 256 (3), 152 (43), 139 (100), 123 (34), 110 (4), 94 (7), 55 (10). ^{1}H NMR (CDCl$_3$, 400 MHz) δ 7.04 (1H, d, *J* = 1.9 Hz, H-2′), 6.83 (1H, dd, *J* = 8.1, 1.9 Hz, H-6′), 6.77 (1H, d, *J* = 8.1 Hz, H-5′), 6.01 (1H, d, H-6), 5.91 (1H, d, *J* = 2.2 Hz, H-8), 4.87 (1H, brs, H-2), 4.20 (1H, m, H-6), 2.85 (1H, dd, *J* = 16.6, 4.4 Hz, H-4b), 2.73 (1H, dd, *J* = 16.6, 3.3 Hz, H-4a); ^{13}C NMR (CDCl$_3$, 100 MHz) δ 79.3 (d, C-2), 66.9 (d, C-3), 28.9 (t, C-4), 157.5 (s, C-5), 96.1 (d, C-6), 157.5 (s, C-7), 95.5 (d, C-8), 157.1 (s, C-9), 99.7 (s, C-10), 132.2 (s, C-1′), 115.4 (d, C-2′), 145.3 (s, C-3′), 145.2 (s, C-4′), 115.2 (d, C-5′), 119.3 (d, C-6′). These data were consistent with those reported[10].

β-Amyrin (11) Colorless needles, EI-MS *m/z* 426 [M]$^+$ (49), 411 (15), 257 (12), 247 (11), 229 (11), 218 (100), 203 (68), 189 (51), 176 (29), 161 (28), 147 (32), 135 (57), 121 (49), 109 (58), 95 (68), 81 (57), 69 (64), 55 (50). ^{1}H NMR (CDCl$_3$, 400 MHz) δ 5.19 (1H, t, *J* = 3.6 Hz, H-12), 1.04, 1.02, 0.96, 0.87, 0.84, 0.82, 0.79 (each 3H, s, Me × 8); ^{13}C NMR (CDCl$_3$, 100 MHz) δ 38.7 (t, C-1), 27.0 (t, C-2), 79.1 (d, C-3), 33.8 (s, C-4), 55.3 (d, C-5), 18.9 (t, C-6), 32.7 (t, C-7), 39.9 (s, C-8), 47.7 (d, C-9), 37.0 (s, C-10), 23.6 (t, C-11), 121.8 (d, C-12), 145.2 (s, C-13), 41.8 (s, C-14), 28.4 (t, C-15), 26.2 (t, C-16), 32.5 (s, C-17), 47.3 (d, C-18), 49.6 (t, C-19), 31.1 (s, C-20), 34.8 (t, C-21), 37.2 (t, C-22), 28.1 (q, C-23), 15.6 (q, C-24), 15.5 (q, C-25), 16.9 (q, C-26), 20.6 (q, C-27), 27.3 (q, C-28), 33.3 (q, C-29), 23.7 (q, C-30). The data were identical to those of reported[11].

Stigmast-5-en-3β, 7α-diol (12) White needles, EI-MS *m/z* 430 [M]$^+$ (25), 412 (100), 398 (35), 271 (8), 252 (7), 229 (6), 211 (6), 175 (8), 161 (12), 147 (11), 135 (15), 109 (10), 93 (13), 81 (19), 69 (21), 55 (35). ^{1}H NMR (CDCl$_3$, 400 MHz) δ 5.58 (1H, dd, *J* = 6.5, 1.8 Hz, H-6), 3.83 (1H, *brs*, H-7), 3.56 (1H, m, H-3), 1.03 (3H, s, Me), 0.90 (3H, d, *J* = 6.4 Hz, Me), 0.82 (3H, t, *J* = 7.8 Hz, Me), 0.78 (3H, d, *J* = 4.4 Hz, Me), 0.77 (3H, d, *J* = 6.7 Hz, Me), 0.66 (3H, s, Me); ^{13}C NMR (CDCl$_3$, 100 MHz) δ 37.1 (t, C-1), 31.4 (t, C-2), 71.4 (d, C-3), 42.1 (t, C-4), 46.3 (s, C-5), 123.9 (d, C-6), 65.4 (d, C-7), 37.6 (d, C-8), 42.2 (d, C-9), 37.3 (s, C-10), 20.8 (t, C-11), 39.2 (t, C-12), 42.3 (s, C-13), 49.5 (d, C-14), 24.3 (t, C-15), 29.3 (t, C-16), 55.8 (d, C-17), 11.7 (q, C-18), 19.1 (q, C-19), 36.1 (d, C-20), 18.3 (q, C-21), 34.0 (t, C-22), 28.3 (t, C-23), 45.9 (d, C-24), 29.3 (d, C-25), 18.8 (q, C-26), 19.8 (q, C-27), 23.1 (t, C-28), 12.0 (q, C-29). The data were equal to those of literature[12].

Scopoletin (**13**), β-sitosterol (**14**) and daucosterol (**15**) were respectively identified by TLC with authentic samples.

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