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紫金龙的生物碱成分研究

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摘　要:从云南白族药物紫金龙(*Dactylicapnos scandens*)中分离得到了11个生物碱,分别鉴定为6-acetonylsanguinarine(**1**)、7-hydroxy-dehydroglaucine(**2**)、demethylsonodione(**3**)、dihydrosanguinarine(**4**)、isocorydione(**5**)、glaucine(**6**)、N-methylisocorydine(**7**)、isocorydine(**8**)、protopine(**9**)、magnoflorine(**10**)和haitinosporine(**11**)。其中化合物**1**～**5**, **7**, **10**～**11**为首次从该植物中分离得到。

关键词:紫金龙;生物碱;化学成分

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Alkaloids from Dactylicapnos scandens

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Abstract: Eleven alkaloids were isolated from the ethanol extract of *Dactylicapnos scandens* and characterized as 6-acetonylsanguinarine(**1**), 7-hydroxy-dehydroglaucine(**2**), demethylsonodione(**3**), dihydrosanguinarine(**4**), isocorydione(**5**), glaucine(**6**), N-methylisocorydine(**7**), isocorydine(**8**), protopine(**9**), magnoflorine(**10**) and haitinosporine(**11**), on the basis of spectroscopic analysis and comparison of their spectral data with those reported. Among them, compounds **1-5**, **7**, **10-11** were isolated from this plant for the first time.

Key words: *Dactylicapnos scandens*; alksloids; chemical constituent

Introduction

The Bai folk drug 'Zi Jin Long' is the dried roots of *Dactylicapnos scandens* (D. Don) Hutch, which mainly grows at a certain altitude of between 1500 and 1800 meters in the Chinese provinces Yunnan and Guangxi [1]. It is used in the treatment of hypertension, inflammation, blooding and pain [2]. Eight alkaloids from the plant have been reported so far [3,4]. During our search for bioactive entities from indigenous Yunnan herbs, we undertook the chemical study on the whole plants of *D. scandens*, and eleven alkaloids were isolated. They were identified as 6-acetonylsanguinarine(**1**), 7-hydroxy-dehydroglaucine(**2**), demethylsonodione(**3**), dihydrosanguinarine(**4**), isocorydione(**5**), glaucine(**6**), N-methylisocorydine(**7**), isocorydine(**8**), protopine(**9**), magnoflorine(**10**) and haitinosporine(**11**) by their spectral data and comparison of their spectral data with those reported. Compounds **1-5**, **7** and **10-11** were isolated from this plant for the first time.

Material and Methods

General

Column chromatography: silica gel(200-300 mesh; H, Qingdao Marine Chemical Inc., Qingdao, P. R. of China); Sephadex LH-20(Amershan Bioscience); reverse-phase C-8 silica gel(40-63 μm, Merck, Darmstadt, German). Melting points were determined on aXRC-1 apparatus and uncorrected. Optical rotations were carried out on a Jasco P-1020 polarimeter. UV spectra were obtained on a Shimadzu UV 2401 PC spectrometer; and MS spectra on a VG Auto spec-3000. NMR spectra were recorded on a Bruker AM-400(400 MHz/100 MHz) spectrometer and chemical shifts were given

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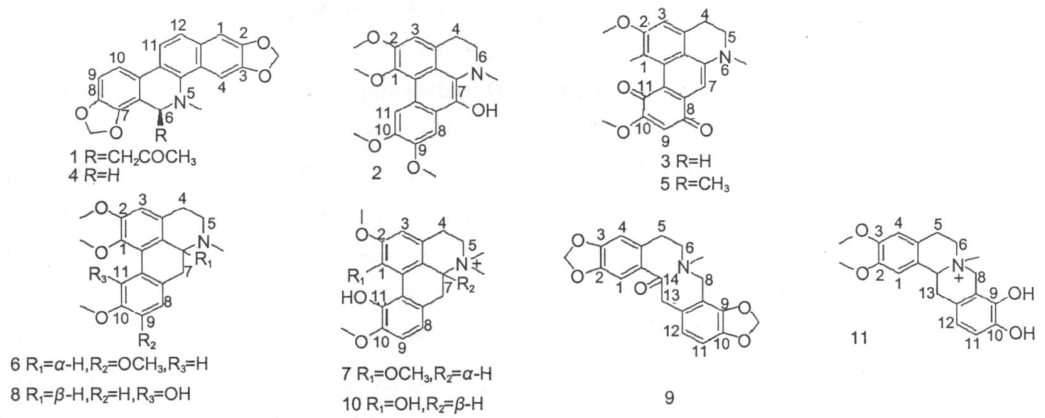

Fig. 1 Compounds 1-11 from Dactylicapnos scandens

in δ with TMS as internal reference. Fractions were monitored by TLC and spots were visualized by spraying the silica gel plates with Dragendorff's reagent.

Plant material

The roots of *D. scandens* were purchased at Lijiang Prefecture, Yunnan Province. The identify of plant material was verified by Prof. Zhou Jun. The specimen (ZJ 2006-09-2) was deposited at the State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, Chinese Academy of Sciences.

Extraction and isolation

The dried roots of *Dactylicapnos scandens* (D. Don) Hutch (4.5 kg) were powered and refluxed in 90% EtOH (10 L) for 3 ×3 h. After removal of the solvent by evaporation, the concentrated extract was suspended in water and extracted successively with chloroform and n-butanol. The chloroform extract (300 g) was divided into two fractions A and B by dry column chromatography (DCC) eluted with EtOAc-MeOH (5 ∶1) on silica gel. Fraction A (63.0 g) was submitted to DCC with petrol-Me_2CO (3 ∶1) to give four fractions A1-A4. Fraction A1 (13.8 g) was subjected to DCC on silica gel with petrol-EtOAc (10 ∶1) to obtain three fractions A1-1-A1-3. Recrystallization of A1-1 in CH_3Cl-Me_2CO (1 ∶1) yielded compound **1** (224 mg). Fraction A1-2 was subjected to reverse phrase column chromatography on Rp-8 eluted with Me_2CO-H_2O (1 ∶1-9 ∶1) to give compounds **2** (23 mg) and **3** (5 mg). Compound **4** (224 mg) was recrystallized from A2 (9.85 g) in Me_2CO.

Fraction A3 VLC on silica gel H eluted with petrol-Me_2CO (4 ∶1-1 ∶1) to give two fractions A4-1 and A4-2. Fraction A4-1 (3.65 g) was subjected to vacuum liquid chromatography (VLC) on silica gel H eluted with petrol-Me_2CO (2 ∶1-1 ∶1) to give two fractions A3-1 and A3-2. Fraction A3-1 was submitted to column chromatography on the Sephadex LH-20 eluted with $CHCl_3$-CH_3OH (1 ∶1) to give compounds **5** (17 mg) and **6** (5 mg). Fraction A4 was subjected to was submitted to VLC on silica gel H eluted with petrol-EtOAc (4 ∶1-1 ∶1) and to reverse phrase column chromatography on Rp-8 eluted with Me_2CO-H_2O (1 ∶1) to give compound **7** (17 mg). Compound **8** (625 mg) was obtained from recrystallization of fraction B from $CHCl_3$-Me_2CO (1 ∶1).

The *n*-butanol extract (100 g) was divided into three fractions C-E by DCC on silica gel eluted with $CHCl_3$-MeOH-H_2O (7 ∶3 ∶0.5). Compound **9** (2.05 g) was obtained from fraction C (10.9 g) by recrystallization from $CHCl_3$-MeOH (1 ∶1). Fraction D was submitted to VLC on silica gel H eluted with $CHCl_3$-EtOH-EtOAc-H_2O (4 ∶4 ∶2 ∶1), reverse phrase column chromatography on Rp-8 eluted with CH_3OH-H_2O (0 ∶1-1 ∶1) and Sephadex LH-20 eluted with CH_3OH-H_2O (8 ∶2) successively to give compounds **10** (24 mg) and **11** (8 mg).

Structure identification

6-Acetonylsanguinarine (1) Colorless needles ($CHCl_3$), mp. 195-197 °C, $[\alpha]_D^{17}$ +20.3 °($CHCl_3$, 0.15). EI-MS *m/z* (%): 389 (18), 333 (25), 332

(100). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.68 (1H, d, *J* = 8.9 Hz, H-10), 7.50 (1H, s, H-4), 7.38 (1H, d, *J* = 8.9 Hz, H-11), 7.13 (1H, s, H-1), 6.88 (1H, d, *J* = 8.9 Hz, H-9), 6.80 (1H, d, *J* = 8.9 Hz, H-12), 6.10 (2H, s, H-20), 6.08 (2H, s, H-19), 4.92 (1H, m, H-6), 2.65 (3H, s, NCH_3), 2.64 (1H, dd, *J* = 15.9, 3.6 Hz, H-1a′), 2.38 (1H, br m, H-1b′), 2.07 (3H, s, H-3′); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 104.3 (C-1), 142.2 (C-2), 147.2 (C-3), 99.8 (C-4), 54.1 (C-6), 148.1 (C-7), 147.6 (C-8), 116.7 (C-9), 107.7 (C-10), 124.2 (C-11), 120.1 (C-12), 138.7 (C-13), 115.6 (C-14), 125.1 (C-15), 130.9 (C-16), 126.9 (C-17), 123.2 (C-18), 101.4 (C-19), 101.7 (C-20), 42.9 (NCH_3), 46.6 (C-1′), 206.3 (C-2′), 30.2 (C-3′). The MS and NMR spectral data were in consistent with those reported [5].

7-Hydroxy-dehydroglaucine (2) Green needles ($CHCl_3$), mp. 240-242 ℃. FAB-MS *m/z* (%): 370 (M^+, 100). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.10 (1H, s, H-11), 7.06 (1H, s, H-8), 6.97 (1H, s, H-3), 6.60 (1H, s, OH), 3.90 (3H, s, OMe-1), 4.02, 4.03, 4.05 (each 3H, s, 3 × OCH_3), 3.36 (2H, m, H-5), 3.26 (2H, m, H-4), 3.06 (3H, s, NCH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 149.1 (C-1), 150.6 (C-2), 110.5 (C-3), 31.2 (C-4), 130.2 (C-4a), 50.5 (C-5), 129.5 (C-6a), 142.2 (C-7), 118.2 (C-7a), 109.1 (C-8a), 145.9 (C-9), 144.6 (C-10), 106.6 (C-11), 101.4 (C-11a), 118.5 (C-11b), 125.3 (C-11c), 55.5, 55.7, 56.3, 59.9 (4 × OCH_3), 40.4 (NCH_3). The MS and NMR spectral data were in consistent with those reported [6].

Demethylsonodione (3) Green prisms (CH_3OH), mp. 230-232 ℃. EI-MS *m/z* (%): 339 (45), 338 (100). ^{1}H NMR (400 MHz, CD_3OD) δ: 7.02 (1H, s, H-7), 6.97 (1H, s, H-3), 5.91 (1H, s, H-9), 3.94 (3H, s, OCH_3-2), 3.89 (3H, s, OCH_3-10), 3.64 (2H, m, H-5), 3.32 (3H, s, NCH_3), 3.10 (2H, m, H-4); ^{13}C NMR (100 MHz, CD_3OD) δ: 140.6 (C-1), 154.6 (C-2), 112.5 (C-3), 127.1 (C-3a), 29.1 (C-4), 51.2 (C-5), 40.8 (NCH_3), 151.1 (C-6a), 103.9 (C-7), 135.8 (C-7a), 187.4 (C-8), 105.5 (C-9), 165.7 (C-10), 177.4 (C-11), 120.0 (C-11a), 125.2 (C-11b), 125.2 (C-11c), 57.4 (OCH_3-2), 56.8 (OCH_3-10). The MS and NMR spectral data were in consistent with those reported [7].

Dihydrosanguinarine (4) Colorless crystals ($CHCl_3$), mp. 195-196 ℃. FAB-MS *m/z* (%): 334 (M^+, 100). ^{1}H NMR (400 MHz, C_5D_5N) δ: 7.87 (1H, d, *J* = 8.0 Hz, H-11), 7.85 (1H, s, H-4), 7.62 (1H, d, *J* = 8.0 Hz, H-12), 7.45 (1H, d, *J* = 8.0 Hz, H-10), 7.30 (1H, s, H-1), 6.98 (1H, d, *J* = 8.0 Hz, H-9), 6.06 (2H, s, OCH_2O-2, 3), 6.03 (2H, s, OCH_2O-7, 8), 4.28 (2H, s, H-6), 2.56 (3H, s, NCH_3); ^{13}C NMR (100 MHz, C_5D_5N) δ: 104.8 (C-1), 148.8 (C-2), 148.2 (C-3), 101.1 (C-4), 127.0 (C-4a), 142.9 (C-4b), 48.7 (C-6), 113.9 (C-6a), 145.2 (C-7), 147.8 (C-8), 107.7 (C-9), 116.8 (C-10), 124.9 (C-10a), 127.5 (C-10b), 120.9 (C-11), 124.5 (C-12), 131.5 (C-12a), 41.5 (NCH_3), 101.8 (OCH_2O-2, 3), 102.0 (OCH_2O-7, 8). The MS and NMR spectral data were in consistent with those reported [8].

Isocorydione (5) Amorphous violet powder, mp. 205-207 ℃. FAB-MS *m/z* (%): 356 (M^+, 100). ^{1}H NMR (400 MHz, DMSO-d_6) δ: 7.23 (1H, s, H-7), 6.79 (1H, s, H-3), 6.02 (1H, s, H-9), 3.51 (2H, m, H-5), 3.31 (3H, s, NCH_3), 3.11 (2H, m, H-4), 3.95 (3H, s, OCH_3-1), 3.91 (3H, s, OCH_3-2), 3.84 (3H, s, OCH_3-10); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 142.8 (C-1), 151.9 (C-2), 112.9 (C-3), 129.1 (C-3a), 28.3 (C-4), 49.5 (C-5), 40.1 (NCH_3), 150.1 (C-6a), 97.1 (C-7), 135.8 (C-7a), 185.4 (C-8), 105.1 (C-9), 163.8 (C-10), 177.4 (C-11) 118.3 (C-11a), 126.1 (C-11b), 116.8 (C-11c), 59.8 (OCH_3-1), 56.5 (OCH_3-2), 56.3 (OCH_3-10). The MS and NMR spectral data were in consistent with those reported [9].

Glaucine (6) Colorless crystals, mp. 120-122 ℃, $[\alpha]_D^{17}$ 110.2 °(EtOH, 0.10). FAB-MS *m/z* (%): 355 (M^+, 100). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.05 (1H, s, H-11), 6.76 (1H, s, H-8), 6.58 (1H, s, H-3), 3.91-3.99 (9H, 3s, OCH_3-2, 9, 10), 3.65 (3H, s, OCH_3-1), 3.30-2.30 (7H, m, C-4, C-5, C-6a, C-7 aliphatic protons), 3.55 (3H, s, NCH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.1 (C-1), 151.7 (C-2), 110.2 (C-3), 128.7 (C-3a), 29.4 (C-4), 53.5 (C-5), 70.1 (C-6a), 35.6 (C-7), 129.2 (C-7a), 110.9 (C-8), 147.8 (C-

9), 147.3 (C-10), 111.9 (C-11), 124.5 (C-11a), 126.9 (C-11b), 126.8 (C-11c), 60.3 (OCH_3-1), 56.4 (OCH_3-2, 9, 10), 44.1 (NCH_3). The MS and NMR spectral data were in consistent with those reported [10].

N-Methylisocorydine (7) Prisms (CH_3OH), mp. 215-217 ℃, $[\alpha]_D^{17}$ 140.2 °(CH_3OH, 0.15). FAB-MS *m/z* (%): 356 (M^+, 100). ^{1}H NMR (400 MHz, CD_3OD) δ: 6.40 (1H, d, *J* = 8.0 Hz, H-8), 6.28 (1H, d, *J* = 8.0 Hz, H-9), 6.25 (1H, s, H-3), 3.51 (3H, s, OCH_3), 3.43 (3H, s, OCH_3), 3.45 (3H, s, OCH_3), 3.37 (1H, br. s, H-6a), 3.05-3.02 (2H, m, H-5), 2.94 (1H, br. d, *J* = 12.4 Hz, H-7ax), 2.91 (3H, s, NCH_3), 2.82-2.25 (2H, m, H-4), 2.52 (1H, br d, *J* = 12.4 Hz, H-7eq), 2.43 (3H, s, NCH_3); ^{13}C NMR (100 MHz, CD_3OD) δ: 142.9 (C-1), 152.9 (C-2), 111.1 (C-3), 129.3 (C-3a), 29.6 (C-4), 51.2 (C-5), 71.4 (C-6a), 31.5 (C-7), 127.5 (C-7a), 119 (C-8), 112.7 (C-9), 149.4 (C-10), 144.3 (C-11), 127.3 (C-11a), 134.3 (C-11b), 112.2 (C-11c), 62.1 (OCH_3-1), 55.9 (OCH_3-2, 10), 55.3, 52.4 (NCH_3). The MS and NMR spectral data were in consistent with those reported [11].

Isocorydine (8) Colorless crystals, mp. 85-87 ℃, $[\alpha]_D^{17}$ -185.2 °($CHCl_3$, 0.05). EI-MS *m/z* (%): 341 (35), 326 (75), mp. 175-178 ℃. ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.66 (1H, s, H-OH), 6.98 (1H, d, *J* = 8.0 Hz, H-8), 6.91 (1H, d, *J* = 8.0 Hz, H-9), 3.87, 3.81 3.67 (9H, 3s, MeO-1, 2, 10), 4.06 (1H, s, H-6a), 3.75-3.46 (2H, m, H-5), 3.38 (1H, m, H-4ax), 3.18 (1H, m, H-7eq), 3.00 (3H, s, NCH_3), 2.59 (1H, s, 7-ax), 2.30 (1H, m, H-4eq); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 144.1 (C-1), 152.8 (C-2), 112.0 (C-3), 127.2 (C-3a), 19.0 (C-4), 51.7 (C-5), 62.1 (C-6a), 32.2 (C-7), 126.6 (C-7a), 119.3 (C-8), 111.7 (C-9), 149.1 (C-10), 144.4 (C-11), 119.2 (C-11a), 123.3 (C-11b), 125.2 (C-11c), 61.6 (CH_3O-1), 56.3 (CH_3O-10), 56.3 (CH_3O-11), 41.4 (NCH_3). Its melting point, MS and NMR were in consistent with those reported [11].

Protopine (9) Colorless crystals (CH_3OH), mp. 205-207 ℃. FAB-MS *m/z* (%): 354 (100). ^{1}H NMR (400 MHz, CD_3OD) δ: 7.04 (1H, s, H-1), 6.65 (1H, s, H-4), 6.88 (1H, d, *J* = 7.6 Hz, H-11), 6.67 (1H, d, *J* = 7.6 Hz, H-12), 4.17 (2H, br. s, H-13), 3.00-3.50 (4H, m, H-5, 6), 6.10 (2H, s, -OCH_2O-2, 3), 6.06 (2H, s, -OCH_2O-9, 10), 2.79 (2H, br. s, H-8), 2.50 (3H, s, NCH_3); ^{13}C NMR (100 MHz, CD_3OD) δ: 108.1 (C-1), 148.0 (C-2), 149.3 (C-3), 102.6 (-OCH_2O-2, 3), 109.7 (C-4), 132.2 (C-4a), 28.0 (C-5), 55.4 (C-6), 41.8 (NCH_3), 54.4 (C-8), 127.4 (C-8a), 146.2 (C-9), 147.8 (C-10), 101.4 (-OCH_2O-9, 10), 111.6 (C-11), 121.3 (C-12), 119.0 (C-12a), 35.0 (C-13), 197.3 (C-14), 132.5 (C-14a). The MS and NMR spectral data were in consistent with those reported [12].

Magnoflorine (10) Yellow prisms (CH_3OH), mp. 242-245 ℃, $[\alpha]_D^{17}$ 195.2 °(CH_3OH, 0.20). FAB-MS *m/z* (%): 342 (M^+, 100). ^{1}H NMR (400 MHz, CD_3OD) δ: 6.39 (1H, d, *J* = 7.7 Hz, H-8), 6.24 (1H, d, *J* = 7.7 Hz, H-9), 6.21 (1H, s, H-3), 3.51 (3H, s, OCH_3), 3.43 (3H, s, OCH_3), 3.37 (1H, br.m, 6a), 3.05 (1H, m, H-5eq), 3.02 (1H, m, H-5ax), 2.94 (1H, br. d, *J* = 12.4 Hz, H-7ax), 2.91 (3H, s, NCH_3), 2.82 (1H, m, H-4ax), 2.52 (1H, br. d, *J* = 12.4 Hz, H-7eq), 2.43 (3H, s, NCH_3), 2.25 (1H, br. d, *J* = 12.8 Hz, H-4eq); ^{13}C NMR (100 MHz, CD_3OD) δ: 152.0 (C-1), 150.9 (C-2), 118.7 (C-3), 118.4 (C-3a), 24.6 (C-4), 62.1 (C-5), 70.5 (C-6a), 31.4 (C-7), 122.4 (C-7a), 111.3 (C-8), 110.2 (C-9), 147.3 (C-10), 147.1 (C-11), 122.0 (C-11a), 121.3 (C-11b), 126.3 (C-11c), 43.6 (NCH_3), 49.9 (NCH_3), 56.3 (OCH_3-2), 56.5 (OCH_3-10). The MS and NMR spectral data were in consistent with those reported [13].

Haitinosporine (11) White prisms (CH_3OH), mp. 242-245 ℃, $[\alpha]_D^{17}$ -110.5 °(CH_3OH, 0.20). FAB-MS *m/z* (%): 342 (M^+, 100). ^{1}H NMR (400 MHz, DMSO-d_6) δ: 7.05 (1H, d, *J* = 8.5 Hz, H-11), 6.87 (1H, d, H-4), 6.85 (1H, d, H-1), 6.83 (1H, d, *J* = 8.5 Hz, H-12), 4.96 (1H, dd, *J* = 8.0, 4.2 Hz, H-C-13a), 4.79 (1H, d, *J* = 15.7 Hz, H-8a), 4.54 (1H, d, *J* = 15.7 Hz, H-8b), 4.00 (1H, m, H-6a), 3.82 (1H, m, H-6b), 3.82 (3H, s, OCH_3-2), 3.80 (1H, m, H-13a), 3.80 (3H, s, OCH_3-3), 3.24 (1H, m, H-5a), 3.16 (1H, m, H-5b), 2.80 (1H, m, H-13b), 2.80

(3H, s, NCH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 111.9 (C-1), 147.8 (C-2), 145.9 (C-3), 112.6 (C-4), 122.8 (C-4a), 22.8 (C-5), 60.6 (C-6), 38.5 (NCH_3), 60.8 (C-8), 114.4 (C-8a), 142.4 (C-9), 145.8 (C-10), 112.0 (C-11), 119.3 (C-12), 122.1 (C-12a), 27.8 (C-13), 79.2 (C-13a), 120.9 (C-13b), 56.1 (OCH_3-3), 55.7 (OCH_3-2). The MS and NMR spectral data were in consistent with those reported [14].

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