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褐薄小齿菌的化学成分研究

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摘 要:从褐薄小齿菌(*Hydnellum concrescens*)子实体中分离得到9个已知化合物,借助光谱手段,它们的化学结构分别鉴定为:friedelin(**1**),(22E,24R)-麦角甾-5,7,22-三烯-3β-醇(**2**),5α,8α-过氧-(22E,24R)-麦角甾-6,22-二烯-3β-醇(**3**),(22E,24R)-麦角甾-5,22-二烯-3β-羟基-7-酮(**4**),6-甲氧基-cerevisterol(**5**),thelephantin K(**6**),thelephantin I(**7**),thelephantin J(**8**),thelephantin L(**9**)。

关键词:褐薄小齿菌;三萜;甾醇;对联三苯

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Study on the Chemical Constitutes of *Hydnellum concrescens*

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Abstract: Nine known compounds have been isolated from the fruiting bodies of *Hydnellum concrescens*. Their structures were established as friedelin(**1**), (22E,24R)-ergosta-5,7,22-trine-3β-ol(**2**), 5α,8α-epidioxy-(22E,24R)- ergosta-6,22-dien-3β-ol(**3**), (22E,24R)-ergosta-5,22-dien-3β-hydroxyl-7-one(**4**), 6-methoxy-cerevisterol(**5**), thelephantin K(**6**), thelephantin I(**7**), thelephantin J(**8**), thelephantin L(**9**) by spectroscopic methods. All compounds are reported from this fungus for the first time.

Key words: *Hydnellum concrescens*; triterpenoid; sterols; p-terphenyls

Introduction

Hydnellum concrescens, a fungus belonging to the family Hydnaceae, is mainly distributed in the province of Zhejiang, Yunnan of China[1]. As a part of our search for naturally occurring bioactive metabolites of the higher fungi in Yunnan Province, the chemical constituents of the fruiting bodies of *H. concrescens* were investigated and nine known compounds have been isolated, including friedelin(**1**), (22E,24R)- ergosta-5,7,22-trine-3β-ol(**2**), 5α,8α-epidioxy-(22E,24R)- ergosta-6,22-dien-3β-ol(**3**), (22E,24R)- ergosta-5,22-dien-3β-hydroxyl-7-one (**4**), 6-methoxy-cerevisterol (**5**), thelephantin K(**6**), thelephantin I(**7**), thelephantin J (**8**), thelephantin L(**9**).

Experimental

General

NMR spectra were recorded on Bruker AV-400 and DRX-500 spectrometers, δ in ppm, J in Hz. IR spectra were obtained with a Bruker Tensor 27 FT-IR with KBr pellets. EI-MS spectra were recorded with a VG Autospec-3000 spectrometer, m/z (rel. int.). HR-ESI-MS was recorded with an API QSTAR Pulsar 1 spectrometer.

Material

Column chromatography was carried out on silica gel (200-300 mesh, Qingdao Marine Chemical Ltd., Qing-

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dao, China) and Sephadex LH-20 (Amersham Biosciences, Uppsala, Sweden).

Fungal material

The fresh fruiting bodies of *H. concrescens* were collected from Wuding of Yunnan Province in China, in August 2005. The voucher specimen was deposited at the herbarium of Kunming Institute of Botany, Chinese Academy of Sciences.

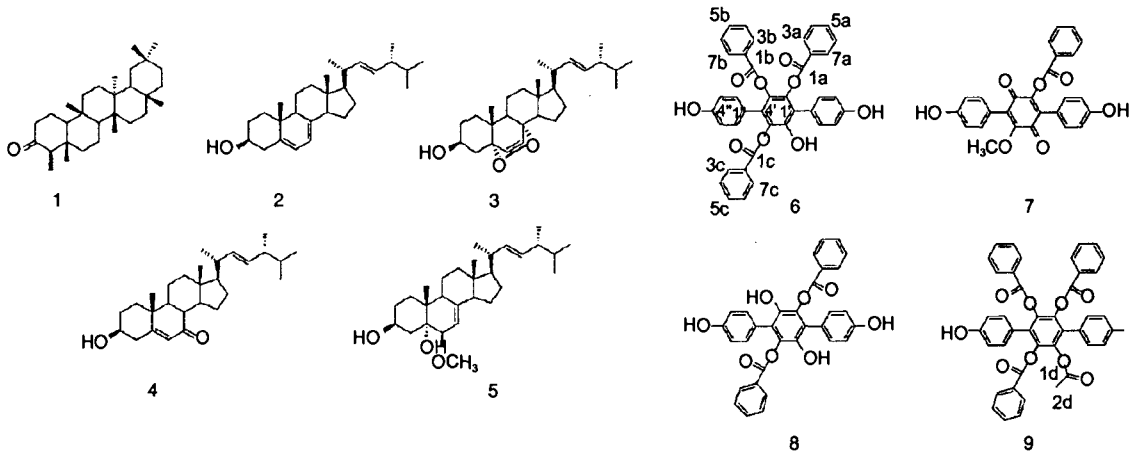

Fig. 1 Structures of compounds 1-9

Extraction and isolation

The fresh fruiting bodies of *H. concrescens* (dry wt. 570 g) were extracted three times with acetone and then three times with $CHCl_3$/MeOH (1∶1, v/v) at r. t. The combined extracts was concentrated *in vacuo* to give a deep brown gum (40 g), which was subjected to column chromatography (silica gel) with a gradient elution of $CHCl_3$/MeOH (100∶0, 98∶2, 95∶5, 90∶10, 80∶20, 50∶50, 0∶100 (v/v)), subsequently seven fractions (A-G) was obtained. Compounds **1** and **2** were obtained from fraction A as needles. Fraction B was rechromatographed over silica gel using petroleum ether/acetone (95∶5) to afford compounds **3-5**. Fraction C was rechromatographed over silica gel using petroleum ether/acetone (5∶1), Sephadex LH-20 ($CHCl_3$/MeOH, 1∶1), Pre-TLC (petroleum ether/acetone, 3∶2) and RP-8 (80% MeOH-H_2O) yielded compound **7**, further purified by repeated CC (silica gel; petroleum ether/acetone, 2∶1) to afford compound **9**. Fraction D was further purified by repeated CC (silica gel; petroleum ether/acetone, 2∶1) to afford compound **6**, then subjected to Sephadex LH-20 (Acetone) yielded compound **8**.

Results and Discussion

Friedelin (1) White needles (acetone), mp. 260-263 ℃, $[\alpha]_D^{20}$ -22.5 ℃ (*c* 1.00, $CHCl_3$); EI-MS *m/z* (%): 426 $[M]^+$ (15), 411 (8), 341 (6), 302 (16), 273 (38), 273 (38), 218 (31), 205 (42); IRυ_{max} cm^{-1}: 1702 (CO); ^{1}H NMR (500 MHz, CD_3COCD_3) δ: 2.27 (1H, m), 1.76-1.31 (22H, m), 1.27 (3H, s), 1.07 (3H, s), 1.03 (3H, s), 1.02 (3H, s), 0.97 (3H, s), 0.90 (3H, s), 0.89 (3H, s), 0.74 (3H, s); ^{13}C NMR (125 MHz, CD_3COCD_3) δ: 22.3 (t, C-1), 41.5 (t, C-2), 213.5 (s, C-3), 58.2 (d, C-4), 42.1 (s, C-5), 41.3 (t, C-6), 18.2 (t, C-7), 53.1 (d, C-8), 37.5 (s, C-9), 59.5 (d, C-10), 35.6 (t, C-11), 30.5 (t, C-12), 39.7 (t, C-13), 38.3 (s, C-14), 32.4 (t, C-15), 36.0 (t, C-16), 30.0 (s, C-17), 42.8 (d, C-18), 35.4 (t, C-19), 28.2 (s, C-20), 32.8 (t, C-21), 39.2 (t, C-22), 6.8 (q, C-23), 14.6 (q, C-24), 17.9 (q, C-25), 20.2 (q, C-26), 18.6 (q, C-27), 32.1 (q, C-28), 35.0 (q, C-29), 31.8 (q, C-30) [2].

(22E,24R)-Ergosta-5,7,22-trien-3β-ol (2) Colorless needles (acetone), mp. 152-154 ℃, $[\alpha]_D^{20}$ -129 ℃ (*c* 0.22, $CHCl_3$); EI-MS *m/z* (%): 396 $[M]^+$ (10), 363 (12), 337 (6), 253 (5), 137 (100), 69 (42); IRυ_{max} cm^{-1}: 3340, 1610; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 5.56 (1H, m), 5.18 (1H, m), 5.18 (2H, m, H-22, 23), 3.62 (1H, m, H-3), 1.02 (3H, d, *J* = 6.8 Hz, H-4), 0.91 (3H, s, H-19), 0.90 (3H, d, *J* = 6.4 Hz, H-

21), 0.78 (3H, d, J = 6.4 Hz, H-26, 27), 0.67 (3H, s, H-18); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 38.4 (t, C-1), 32.0 (t, C-2), 70.5 (d, C-3), 40.8 (t, C-4), 139.8 (s, C-5), 119.6 (d, C-6), 116.3 (d, C-7), 141.3 (s, C-8), 46.3 (d, C-9), 37.1 (s, C-10), 21.1 (t, C-11), 39.1 (t, C-12), 42.8 (s, C-13), 54.6 (d, C-14), 23.0 (t, C-15), 28.3 (t, C-16), 55.8 (d, C-17), 12.1 (q, C-18), 16.3 (q, C-19), 40.4 (d, C-20), 21.1 (q, C-21), 135.6 (d, C-22), 132.0 (d, C-23), 40.8 (d, C-24), 33.1 (q, C-25), 20.0 (d, C-26), 19.6 (q, C-27), 17.6 (q, C-28)[3].

5α,8α-Epidioxy-(22E,24R)-ergosta-6,22-dien-3β-ol (3) White needles (acetone), mp. 177-178 ℃, $[\alpha]_D^{20}$ +20 ℃ (c 0.10, $CHCl_3$); EI-MS m/z (%): 428 $[M]^+$ (13), 410 $[M-H_2O]^+$ (5), 396 $[M-O_2]^+$ (100), 363 (35), 303 (8), 251 (20), 152 (30), 107 (24), 95 (35), 81 (43), 69 (65); IRυ_{max} cm^{-1}: 3525, 3309, 2955, 1650, 1380, 1074; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 6.49 (1H, d, J = 8.5 Hz), 6.23 (1H, d, J = 8.5 Hz), 5.20 (1H, dd, J = 7.6, 7.5 Hz), 5.12 (1H, dd, J = 7.6, 7.5 Hz), 3.94 (1H, m), 2.08-1.49 (20H, m), 1.20 (3H, s), 0.98 (3H, d, J = 6.6 Hz), 0.88 (3H, d, J = 6.8 Hz), 0.86 (3H, s), 0.82 (3H, d, J = 3.6 Hz), 0.79 (3H, d, J = 3.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 34.1 (t, C-1), 30.2 (t, C-2), 66.5 (d, C-3), 37.0 (t, C-4), 82.1 (s, C-5), 135.5 (d, C-6), 130.8 (d, C-7), 79.4 (s, C-8), 51.2 (d, C-9), 37.0 (s, C-10), 23.4 (t, C-11), 39.4 (t, C-12), 44.6 (s, C-13), 51.7 (d, C-14), 20.6 (t, C-15), 28.6 (t, C-16), 56.3 (d, C-17), 12.9 (q, C-18), 18.2 (q, C-19), 39.7 (d, C-20), 20.9 (q, C-21), 135.2 (d, C-22), 132.4 (d, C-23), 42.8 (d, C-24), 33.1 (q, C-25), 19.1 (d, C-26), 19.6 (q, C-27), 17.6 (q, C-28)[4].

(22E, 24R)-Ergosta-5, 22-dien-3β-hydroxy-7-one (4) White needles (acetone), mp. 170-171 ℃, $[\alpha]_D^{20}$ -93 ℃ (c 0.19, $CHCl_3$); EI-MS m/z (%): 412 $[M]^+$ (33), 397 (5), 379 (4), 369 (9), 351 (5), 314 (62), 287 (79), 285 (52), 269 (24), 245 (18), 205 (20), 191 (30), 161 (44), 107 (69), 69 (78), 55 (100); IRυ_{max} cm^{-1}: 3392, 1672; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 5.67 (1H, d, H-6), 5.17 (2H, m, H-22 and H-23), 3.65 (1H, bm, Hax-3), 2.48 (1H, m, Heq-4), 2.35 (1H, m, Hax-4), 2.23 (1H, dd, H-8), 1.94 (1H, m, H-20), 1.82 (1H, m, H-24), 1.44 (1H, m, H-25), 1.17 (3H, s, H-19), 0.99 (3H, d, J = 6.6 Hz, H-21), 0.89 (3H, d, J = 6.8 Hz, H-28), 0.81 (3H, d, J = 6.7 Hz, H-26 or H-27), 0.79 (3H, d, J = 6.8 Hz, H-27 or H-26), 0.67 (3H, s, H-18). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 36.3 (t, C-1), 31.0 (t, C-2), 70.4 (d, C-3), 41.8 (t, C-4), 165.6 (s, C-5), 125.9 (d, C-6), 202.6 (s, C-7), 45.3 (d, C-8), 50.0 (d, C-9), 38.5 (s, C-10), 21.1 (t, C-11), 38.5 (t, C-12), 43.0 (s, C-13), 49.9 (d, C-14), 26.3 (t, C-15), 28.6 (t, C-16), 54.7 (d, C-17), 12.2 (q, C-18), 17.3 (q, C-19), 39.9 (d, C-20), 21.1 (q, C-21), 135.6 (d, C-22), 131.9 (d, C-23), 42.8 (d, C-24), 33.1 (d, C-25), 17.6 (q, C-26), 19.6 (q, C-27), 20.0 (q, C-28)[5].

6-Methoxy-cerevisterol (5) White needles (acetone), mp. 237-238 ℃, $[\alpha]_D^{20}$-104 ℃ (c 0.15, $CHCl_3$); EI-MS m/z (%): 444 $[M]^+$ (2), 426 $[M-H_2O]^+$ (100), 411 (40), 393 (47), 377 (32), 355 (20), 337 (30), 283 (24), 269 (12), 251 (20); IRυ_{max} cm^{-1}: 3540, 2955, 2812, 1465; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 5.37 (1H, dd, J = 5.1, 2.6 Hz, H-7), 5.14-5.23 (2H, m, H-22, 23), 4.03 (1H, m, H-3), 3.37 (3H, s, CH_3), 3.14 (1H, bd, J = 5.1 Hz, H-6), 1.20-2.10, 1.06 (3H, s, H-19), 1.00 (3H, d, J = 6.6 Hz, H-21), 0.89 (3H, d, J = 6.9 Hz, H-28), 0.81 (3H, d, J = 6.6 Hz, H-26), 0.80 (3H, d, J = 6.5 Hz, H-27), 0.57 (3H, s, H-18); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 32.7 (C-1), 30.8 (C-2), 67.8 (C-3), 39.6 (C-4), 76.3 (C-5), 82.4 (C-6), 115.0 (C-7), 143.6 (C-8), 42.8 (C-9), 37.2 (C-10), 22.9 (C-11), 39.3 (C-12), 43.8 (C-13), 54.9 (C-14), 22.1 (C-15), 27.9 (C-16), 55.9 (C-17), 12.3 (C-18), 18.3 (C-19), 40.5 (C-20), 21.1 (C-21), 135.4 (C-22), 132.0 (C-23), 43.8 (C-24), 33.1 (C-25), 19.9 (C-26), 19.6 (C-27), 17.6 (C-28)[6].

Thelephantin K (6) Grayish solid; Negative FAB-MS: 637 $[M-H]^-$; IRυ_{max} cm^{-1}: 3447, 1745, 1611, 1526; UVλ_{max}^{MeOH} nm: 231, 260; ^{1}H NMR (500 MHz, DMSO-d_6) δ: 9.50, 9.48, 9.42 (3H, s, 3OH), 7.22 (2H, d, J = 8.4 Hz, H-2, 6), 6.74 (2H, d, J = 8.4 Hz, H-3, 5), 7.38 (1H, d, J = 8.4 Hz, H-2″), 6.77 (1H, d, J = 8.4 Hz, H-3″), 6.62 (1H, d, J = 8.4 Hz, H-5″),

7.21(1H,d,J = 8.4 Hz,H-6″),7.76(2H,d,J = 8.5 Hz,H-3a,7a),7.28(overlap,H-4a,6a),7.44(1H,t,J = 7.7 Hz,H-5a),8.01(1H,d,J = 8.5 Hz,H-3b),7,47(2H,t,J = 8.2 Hz,H-4b,6b),7.59(1H,m,H-5b),7.97(1H,d,J = 8.5 Hz,H-7b),7.76(1H,d,J = 8.5 Hz,H-3c),7.28(overlap,H-4c),7.44(1H,t,J = 7.7 Hz,H-5c),7.28(overlap,H-6c),7.76(1H,d,J = 8.5 Hz,H-7c);^{13}C NMR(125 MHz,DMSO-d_6) δ:124.9(s,C-1),132.6(d,C-2,6),116.0(d,C-3,5),158.0(s,C-4),124.1(s,C-1′),135.0(C-2′),134.8(s,C-3′),124.1(s,C-4′),135.0(s,C-5′),142.7(s,C-6′),124.9(s,C-1″),132.7(d,C-2″),116.1(d,C-3″),158.3(s,C-4″),115.9(d,C-5″),132.0(d,C-6″),166.4(s,C-1a),130.8(s,C-2a),130.7(d,C-3a,7a),129.5(d,C-4a,6a),134.6(d,C-5a),166.3(s,C-1b),129.8(s,C-2b),130.5(d,C-3b),129.5(d,C-4b,6b),134.2(d,C-5b),131.1(d,C-7b),166.4(s,C-1c),130.8(s,C-2c),130.7(d,C-3c),129.5(d,C-4c),134.6(d,C-5c),129.5(C-6c),130.7(d,C-7c)[7,8].

Thelephantin I(7) Red powder;Negative FAB-MS:441 [M-H]$^-$;IRυ_{max} cm^{-1}:3376,1740,1661,1606,1513;UVλ_{max}^{MeOH} nm:242,260,248;^{1}H NMR(500 MHz,CD_3COCD_3)δ:8.81,8.68(2H,s,2OH),7.36(2H,d,J = 8.6 Hz,H-2,6),6.89(2H,d,J = 8.6 Hz H-3,5),7.25(1H,d,J = 8.6 Hz,H-2″,6″),6.86(1H,d,J = 8.4 Hz,H-3″,5″),8.04(2H,d,J = 7.3 Hz,H-3a,7a),7.55(2H,H-4a,6a),7.71(1H,t,J = 7.3 Hz,H-5a),3.93(3H,s,CH_3O);^{13}C NMR(125 MHz,CD_3COCD_3)δ:120.8(s,C-1),132.8(d,C-2,6),115.9(d,C-3,5),159.5(s,C-4),129.3(s,C-1′),148.3(C-2′),184.3(s,C-3′),129.2(s,C-4′),156.3(s,C-5′),182.2(s,C-6′),122.0(s,C-1″),133.0(d,C-2″),115.7(d,C-3″),158.7(s,C-4″),115.7(d,C-5″),133.0(d,C-6″),165.7(s,C-1a),134.8(s,C-2a),130.9(d,C-3a,7a),129.7(d,C-4a,6a),135.1(d,C-5a),61.6(s,CH_3O)[7,8].

Thelephantin J(8) Grayish solid;Negative FAB-MS:533 [M-H]$^-$;IRυ_{max} cm^{-1}:3258,1720,1610,1526;UVλ_{max}^{MeOH} nm:231,262;^{1}H NMR(400 MHz;CD_3OD)δ:7.27(2H,d,J = 8.8 Hz,H-2,6),6.75(2H,d,J = 8.8 Hz,H-3,5),7.27(2H,d,J = 8.8 Hz,H-2″,6″),6.75(1H,d,J = 8.8 Hz,H-3″,5″),7.74(2H,dd,J = 1.4,8.5 Hz,H-3a,7a),7.26(2H,t,J = 8.5 Hz,H-4a,6a),7.43(1H,t,J = 8.5 Hz,H-5a),7.74(2H,d,J = 8.5 Hz,H-3c,7c),7.26(2H,t,J = 8.5 Hz,H-4c,6c),7.43(1H,t,J = 8.5 Hz,H-5c);^{13}C NMR(100 MHz,CD_3OD)δ:124.5(s,C-1),132.3(d,C-2,6),115.8(d,C-3,5),157.7(s,C-4),124.3(s,C-1′),132.3(C-2′),141.9(s,C-3′),124.3(s,C-4′),132.3(s,C-5′),141.9(s,C-6′),124.5(s,C-1″),132.3(d,C-2″),115.8(d,C-3″),157.7(s,C-4″),115.9(d,C-5″),132.3(d,C-6″),166.9(s,C-1a),130.3(s,C-2a),130.4(d,C-3a,7a),129.5(d,C-4a,6a),134.7(d,C-5a),165.0(s,C-1c),130.2(s,C-2c),130.7(d,C-3c),129.5(d,C-4c),134.7(d,C-5c),129.5(d,C-6c),130.4(d,C-7c)[7,8].

Thelephantin L(9) Grayish solid;Positive FAB-MS:703 [M + Na]$^+$;IRυ_{max} cm^{-1}:3452,1748,1611,1525;UVλ_{max}^{MeOH} nm:232,267;^{1}H NMR(500 MHz,CD_3OD)δ:8.48,8.38(2H,s,2OH),7.22(2H,d,J = 8.8 Hz,H-2,6),6.75(2H,d,J = 8.8 Hz H-3,5),7.24(1H,d,J = 8.8 Hz,H-2″,6″),6.65(1H,d,J = 8.8 Hz,H-3″,5″),7.79(2H,d,J = 8.2 Hz,H-3a,7a),7.28(t,J = 8.2 Hz,H-4a,6a),7.47(1H,t,J = 8.2 Hz,H-5a),7.79(1H,d,J = 8.2 Hz,H-3b),7.28(2H,t,J = 8.2 Hz,H-4b,6b),7.47(1H,t,J = 8.2 Hz,H-5b),7.79(1H,d,J = 8.2 Hz,H-7b),7.97(1H,d,J = 8.5 Hz,H-3c),7.49(1H,t,J = 8.5 Hz,H-4c),7.64(1H,t,J = 8.5 Hz,H-5c),7.49(1H,t,J = 8.5 Hz,H-6c),7.97(1H,d,J = 8.5 Hz,H-7c),2.00(3H,s,CH_3CO);^{13}C NMR(125 MHz,CD_3OD)δ:123.7(s,C-1),131.7(d,C-2,6),116.0(d,C-3,5),158.0(s,C-4),131.8(s,C-1′),140.9(C-2′),140.9(s,C-3′),131.8(s,C-4′),140.9(s,C-5′),140.9(s,C-6′),123.7(s,C-1″),131.7(d,C-2″),116.0(d,C-3″),158.0(s,C-4″),116.0(d,C-5″),131.7(d,C-6″),164.9(s,C-1a),129.3(s,C-2a),130.8(d,C-3a,7a),129.6(d,C-4a,6a),134.4(d,C-5a),164.9(s,C-1b),129.3(s,C-2b),130.8(d,C-3b),129.6(d,C-4b,6b),134.4(d,C-5b),130.8(d,C-7b),164.9(s,C-1c),129.3(s,C-2c),131.4(d,C-3c),130.3(d,C-4c),135.3(d,C-5c),129.6(C-6c),130.8(d,C-7c),169.6(s,C-1d),20.0(q,C-2d)[7,8].

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