文章编号:1001-6880(2014)7-1034-05

玉竹的化学成分研究

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摘　要:采用硅胶、Sephadex LH-20 等多种材料进行分离纯化,通过理化方法和波普分析进行结构鉴定,从玉竹根茎的醇提溶液的乙酸乙酯萃取部分中分离鉴定了 12 个化合物,分别为:ningpogenin (**1**)、6-*O*-*p*-hydroxybenzoylaucubin (**2**)、3,3′-Bisdemethylpinoresinol (**3**)、5-Hydroxymethyl-2-furancarboxaldehyde (**4**)、(22*E*,24*R*)-Ergosta-7,22-dien-3β-ol(**5**)、Borreriagenin (**6**)、麦角甾-7,22-二烯-3β,5α,6β-三醇 (**7**)、5α,8α-epidiory-(22*E*,24*R*)-ergosta-6,22-dien-3β-ol (**8**)、β-谷甾醇 (**9**)、3β,5α,9α-三羟基-麦角甾-7,22-二烯-6-酮 (**10**)、麦角甾-7,22-二烯-3-酮 (**11**)、对羟基苯乙醇 (**12**)。除化合物(**9**)之外,其余所有化合物均为首次从该植物中分离得到。

关键词:玉竹;提取;分离;结构鉴定

中图分类号:O629.9　　**文献标识码**:A

Study on Chemical Constituents of *Polygonatum odoratum*

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Abstract: Twelve compounds were separated and purified from the ethyl acetate extracts in the alcohol solution of *Polygonatum* rhizome by silica gel and Sephadex LH-20 column chromatography, respectively. Their structures were identified as ningpogenin (**1**); 6-*O*-*p*-hydroxybenzoylaucubin (**2**); 3,3′-Bisdemethylpinoresinol (**3**); 5-Hydroxymethyl-2-furancarboxaldehyde (**4**); (22*E*,24*R*)-Ergosta-7,22-dien-3β-ol (**5**); Borreriagenin (**6**); Cerevisterol (**7**); 5α,8α-epidiory-(22*E*,24*R*)-ergosta-6,22-dien-3β-ol (**8**); β-sitosterol (**9**); 3β,5α,9α-trihydroxyergosta-7-22-dien-6-one (**10**); ergosta-7,22-dien-3-one (**11**); p-hydroxybenzylethanol (**12**). All the compounds were isolated from the plant for the first time, except the compound (**9**).

Key words: *Polygonatum odoratum*; extraction; isolation; structure identification

玉竹(*Polygonatum odoratum* (Mill.) Druce)为百合科(*Liliaceae*)黄精属(*Polygo-natum*)植物,又名玉参,其根茎横走,肉质黄白色,密生多数须根,叶面绿色,下面灰色。花腋生,通常 1 ~ 3 朵簇生,耐寒、阴。具有生津止渴等功效。临床上常用其根茎治疗虚劳发热、咳嗽以及小便频数等症。同时,亦有研究表明[1],其醇提物有增强免疫、抗菌、降压等作用。目前有关玉竹根茎的化学成分的报道较少,本文用乙醇作为提取溶剂,乙酸乙酯萃取,分离鉴定其化合物成分,现报道如下:

1　仪器与材料

熔点由四川大学科学仪器厂生产的 XTC-1 型显微熔点仪测定;红外光谱由瑞士 Brucker 公司产 Bio-Rad FTS-135 型红外光谱仪测定,溴化钾压片;质谱由英国 Micromass 公司产 VG Auto-Spec-3000 质谱仪测定;^{1}H,^{13}C NMR 用 Bruker DRX-500 MHz 超导核磁共振仪测定,TMS 为内标;HPLC 为 Agilent 1100, ZorbaxSB-C_{18} column, 5 μm, 4.6 × 150 mm; Sephadex LH-20 为瑞士 Amershan Biosciences 公司生产;柱层析硅胶和 GF_{254} TLC 预制板均为青岛海洋化工厂生产。

显色方法为 254、365 nm 荧光、10% 硫酸乙醇溶液和硫酸香草醛溶液处理后加热显色、硫酸铜丙酮显色及碘蒸气显色。

玉竹干燥根茎(Polygonatum odoratum (Mill.) Druce),由安徽中医学院刘金旗教授鉴定。

2　提取与分离

玉竹干燥根茎,切至片状(*Polygonatum odora-*

收稿日期:2013-07-04　　接受日期:2013-09-27

基金项目:安徽新华学院校级重点扶持学科建设(zdfcx201103)

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tum,约4.0 kg)用95%乙醇(5×6 L)提取,减压浓缩至无醇味,加水悬浮,用乙酸乙酯(5×4 L)萃取,减压浓缩得浸膏10.2 g。经硅胶柱层析($CHCl_3$-MeOH梯度洗脱:100/0,98/2,95/5,90/10,80/20;v/v),得五个部分(Fr. a~Fr. e)。

Fr. b($CHCl_3$-MeOH = 98∶2的洗脱部分)浓缩后,经多次硅胶柱分离(petroleum ether- Me2CO = 9∶1- 7∶3,petroleum ether-EtOAc = 8∶2- 5∶5,v/v),最后用Sephadex LH-20凝胶柱纯化($CHCl_3$-MeOH = 1∶1,v/v),最终得到化合物**1**(5.0mg)、化合物**3**(8.9 mg)、化合物**4**(10.3 mg)、化合物**5**(6.0 mg)、化合物**10**(9.5mg);Fr. c($CHCl_3$-MeOH = 95∶5的洗脱部分)浓缩后(浅黄色油状物),经凝胶柱($CHCl_3$- MeOH = 1∶1,v/v)纯化和硅胶层析柱(petroleum ether-Me_2CO = 8∶2- 4∶6,v/v)得到化合物**2**(12.3 mg)、化合物**7**(13.5 mg)、化合物**8**(9.5 mg);Fr. d($CHCl_3$-MeOH = 90∶10的洗脱部分)浓缩后(棕色油状物),通过HPLC(CH_3CN- H_2O,20-100%,v/v)分析,经硅胶柱(petroleum ether-EtOAc = 6∶1-3∶1,v/v)洗脱,Sephadex LH-20凝胶柱纯化($CHCl_3$-MeOH = 1∶1,v/v),化合物**6**(6.8 mg)、化合物**9**(14.3 mg)、化合物**11**(10.4mg)、化合物**12**(13.0 mg)。

3 结构鉴定

化合物1 (ningpogenin),$C_9H_{14}O_3$,无色油状,^{1}H NMR(500 MHz,CD_3OD)δ:4.90(1H,m,J = 7.6,1.5,1.3 Hz,H-1),3.62(2H,ddd,J = 8.9,6.8 Hz,H-3),3.73(2H,ddd,J = 9.1,6.0,7.7 Hz,H-4),1.94(2H,m,J = 12.1,7.0,6.0,6.3 Hz,H-4′),1.85(2H,m,J = 1.2,5.8,7.3,9.1 Hz,H-3′),3.02(1H,m,J = 6.2,9.3,7.3,7.5 Hz,H-5),2.94(1H,m,J = 7.5,6.4,7.5,1.5,1.5,1.6,1.4 Hz,H-6),5.7(1H,m,J = 1.3,1.6,1.6,1.7 Hz,H-7),3.78(2H,dd,J = 10.7,6.3 Hz,H-9),3.66(2H,dd,J = 10.5,7.7 Hz,H-9′),4.16(2H,m,J = 1.4,1.5,1.6,1.5 Hz,H-10),4.14(2H,m,J = 1.4,1.5,1.6,1.1 Hz,H-10′);^{13}C NMR(125 MHz,CD_3OD)δ:88.5(C-1),68.1(C-3),28.3(C-4),44.3(C-5),49.8(C-6),150.3(C-7),126.6(C-8),62.8(C-9),60.4(C-10)。以上数据与文献值相一致[2]。确定为:ningpogenin。

化合物2 (6-*O*-*p*-hydroxybenzoylaucubin),$C_{22}H_{26}O_{11}$,无色油状,^{1}H NMR(500 MHz,CD_3OD)δ:5.14(d,5.8Hz,H-1),6.33(dd,J = 6.3 Hz,J = 1.8 Hz,H-3),5.09(dd,J = 6.2 Hz,J = 3.8 Hz,H-4),3.04(m,H-5),5.53(m,H-6),5.88(m,H-7),3.12(br t,J = 6.2 Hz,H-9),4.23(br. d,J = 15.8 Hz,H-10),4.71(d,J = 7.7 Hz,H-1′),3.23(dd,J = 9.3,7.8 Hz,H-2′),3.38(m,H-3′),3.5(obsc,H-4′),3.5(obsc,H-5′),3.89(br. d,J = 12.0 Hz,H-6′),7.85(2H,J = 8.7 Hz,H-2″,H-6″),7.85(2H,J = 8.6Hz,H-3″,H-5″);^{13}C NMR(125 MHz,CD_3OD)δ:96.7(C-1),141.8(C-3),105.3(C-4),42.6(C-5),85.7(C-6),126.6(C-7),151.5(C-8),48.4(C-9),61.6(C-10),99.2(C-1′),74.4(C-2′),77.7(C-3′),72.0(C-4′),78.5(C-5′),62.5(C-6′),122.3(C-1″),132.5(C-2″),116.3(C-3″),163.4(C-4″),116.2(C-5″),132.9(OMe),168.4(CO′)。以上数据与参考文献相一致[3]。确定为:6-*O*-*p*-hydroxybenzoylaucubin。

化合物3 (3,3′-Bisdemethylpinoresinol),$C_{18}H_{18}O_6$,黄色粉末,^{1}H NMR(500 MHz,acetone-d_6)δ:6.88(1H,d,J = 1.8 Hz,H-2,2′),6.81(1H,d,J = 8.1 Hz,H-5,5′),6.72(1H,dd,J = 8.3,1.9 Hz,H-6,6′),4.61(1H,d,J = 4.2 Hz,H-7,7′),3.03(1H,m,H-8,8′),3.79(1H,dd,J = 8.7,6.9 Hz,H-9a,9a′),4.15(1H,dd,J = 8.9,6.9 Hz,H-9b,9b′);^{13}C NMR(125 MHz,acetone-d_6)δ:134.3(s,C-1),114.3(d,C-2),145.1(s,C-3),145.8(s,C-4),115.8(d,C-5),118.6(d,C-6),86.5(d,C-7),54.8(d,C-8),71.9(t,C-9),134.3(s,C-1′),114.3(d,C-2′),145.4(s,C-3′),145.8(s,C-4′),115.8(d,C-5′),118.6(d,C-6′),86.5(d,C-7′),54.9(d,C-8′),72.1(t,C-9′);EI-MS m/z(%):330[M]$^+$(33),299(11),191(17),149(42),137(100)。以上数据与参考文献值相一致[4]。确定为:3,3′-Bisdemethylpinoresinol。

化合物4 (5-Hydroxymethyl-2-furancarboxaldehyde),$C_6H_6O_3$,淡黄色油状物,^{1}H NMR(500 MHz,acetone-d_6)δ:6.53(1H,d,J = 3.5 Hz,H-3),7.27(1H,d,J = 3.3 Hz,H-4),9.57(1H,s,H-6),4.73(2H,s,H-7);^{13}C NMR(125 MHz,acetone-d_6)δ:162.9(s,C-2),110.2(d,C-3),123.5(d,C-4),153.6(s,C-5),177.7(s,C-6),57.8(t,C-7)。以

上数据与文献值相一致[5]。确定为:5-Hydroxymethyl-2-furancarboxaldehyde.

化合物 5 (22*E*,24*R*)-Ergosta-7,22-dien-3β-ol,无色针晶,$C_{28}H_{46}O$,EI-MS *m/z* (%):398 [M]$^+$ (17),383 [M-CH_3]$^+$ (15),273 (20),271 (100),255 (37);^{1}H NMR ($CDCl_3$,500 MHz) δ:3.57 (1H,m,H-3),0.55 (3H,s,H-18),0.79 (3H,s),5.20 (2H,m,H-22,23),0.83 (3H,d,*J* = 6.7 Hz,H-27),0.93 (3H,d,*J* = 6.2 Hz,H-28),0.98 (3H,d,*J* = 6.8 Hz,H-21);^{13}C NMR ($CDCl_3$,125 MHz) δ:37.1 (C-1),31.6 (C-2),71.2 (C-3),38.2 (C-4),40.3 (C-5),29.8 (C-6),117.6 (C-7),139.8 (C-8),49.6 (C-9),34.3 (C-10),21.7 (C-11),39.6 (C-12),43.5 (C-13),55.3 (C-14),22.9 (C-15),28.3 (C-16),56.2(C-17),12.5 (C-18),13.1 (C-19),40.5 (C-20),21.3 (C-21),135.7 (C-22),131.6 (C-23),42.9 (C-24),33.4 (C-25),19.7 (C-26),19.7 (C-27),17.9 (C-28)。以上数据与文献值相一致[6]。确定为:(22*E*,24*R*)-Ergosta-7,22-dien-3β-ol。

化合物 6 Borreriagenin,$C_{10}H_{14}O_5$,白色粉末;^{1}H NMR (500 MHz,acetone-d_6):3.86 (1H,m,H-1a),3.79 (1H,m,H-1b),3.92 (1H,m,H-3a),3.88 (1H,m,H-3b),2.99 (1H,m,H-4),3.36 (1H,m,H-5),5.43 (1H,d,*J* = 7.7 Hz,H-6),5.85 (1H,brs,H-7),2.99 (1H,m,H-9),4.73 (1H,m,H-10a),4.21 (1H,m,H-10b);^{13}C NMR (125 MHz,acetone-d_6):60.9 (t,C-1),62.9 (t,C-3),45.8 (d,C-4),44.1 (d,C-5),88.4 (d,C-6),125.2 (d,C-7),153.5 (s,C-8),48.5 (d,C-9),60.6 (t,C-10),181.1 (s,C-11);EI-MS *m/z* (%):214 [M]$^+$ (1),166 (50),136 (100)。以上数据与文献值相一致[7]。确定为:Borreriagenin.

化合物 7 麦角甾-7,22-二烯-3β,5α,6β-三醇(Cerevisterol),$C_{28}H_{46}O_3$,无色针晶;mp. 253 ~ 255 ℃;^{1}H NMR (C_5D_5N,500 MHz):δ 5.75 (1H,s,H-7),5.23 (1H,dd,15.4,7.3,H-23),5.17 (1H,dd,15.4,8.4,H-22),4.85 (1H,m,H-3),4.33 (1H,br. d,4.9,H-6),1.53 (3H,s,H-19),1.08 (3H,d,6.5,H-21),0.95 (3H,d,6.9,H-28),0.86(3H,d,6.8,H-27),0.85 (3H,d,6.8,H-26),0.68 (3H,s,H-18);^{13}C NMR (C_5D_5N,125 MHz):δ 33.9 (C-1),32.7 (C-2),67.7 (C-3),42.1 (C-4),76.6 (C-5),74.4 (C-6),120.6 (C-7),141.7 (C-8),43.9 (C-9),38.2 (C-10),22.5 (C-11),40.2 (C-12),43.8 (C-13),55.3 (C-14),23.6 (C-15),28.3 (C-16),56.6 (C-17),12.4 (C-18),18.9 (C-19),40.8 (C-20),21.5 (C-21),136.3 (C-22),132.6 (C-23),43.1 (C-24),33.2 (C-25),19.8(C-26),20.2 (C-27),17.7 (C-28);EI-MS *m/z* (%):430 [M] +,412 [M-H_2O]$^+$ (35),394 [M-2H_2O]$^+$ (37),379 (65),376 [M-3H_2O]$^+$ (15),269 (33),251 (62),69 (100)。以上数据与文献值相一致[8]。确定为:麦角甾-7,22-二烯-3β,5α,6β-三醇。

化合物 8 (5α,8α-epidiory-(22*E*,24*R*)-ergosta-6,22-dien-3β-ol),无色针晶,$C_{28}H_{44}O_3$,mp. 176-178 ℃;IR (KBr) (cm^{-1}):3523,3305,2959,2876,1656,1458,1375;EI-MS *m/z* (%):428[M]$^+$ (10),410 (4),396 (100),363 (35),271 (7),251 (14);^{1}H NMR (500 MHz,$CDCl_3$) δ:6.49 (1H,d,*J* = 8.7 Hz,H-6),6.25 (1H,d,*J* = 8.7 Hz,H-7),5.22 (1H,dd,*J* = 7.8,7.7 Hz,H-23),5.15 (1H,dd,*J* = 7.5 Hz,H-22),3.96 (1H,m,H-3),2.07 - 1.49 (20H,m,steroid necleus),0.98 (3H,d,*J* = 6.6 Hz,H-21),0.90 (3H,d,*J* = 6.8 Hz,H-28),0.87 (3H,d,*J* = 3.4 Hz,H-26),0.79 (3H,d,*J* = 3.4 Hz,H-27);^{13}C NMR (125 MHz,$CDCl_3$) δ:34.8 (t,C-1),30.6 (t,C-2),66.6 (d,C-3),37.4 (t,C-4),82.5 (s,C-5),135.9 (d,C-6),130.8 (d,C-7),79.5 (s,C-8),51.7 (d,C-9),37.1 (s,C-10),23.9 (t,C-11),39.4 (t,C-12),45.0 (s,C-13),51.9 (d,C-14),20.7 (t,C-15),28.7 (t,C-16),56.8 (d,C-17),13.0 (q,C-18),18.3 (q,C-19),39.8 (d,C-20),21.0 (q,C-21),135.6 (d,C-22),132.4 (d,C-23),42.6 (d,C-25),33.4 (d,C-25),19.6 (q,C-26),19.2 (q,C-28),17.8(q,C-28)。以上数据与文献值相一致[9]。确定为:5α,8α-epidiory-(22*E*,24*R*)-ergosta-6,22-dien-3β-ol。

化合物 9 β-谷甾醇,$C_{29}H_{50}O$,白色针晶(丙酮);^{1}H NMR (500 MHz,$CDCl_3$):5.33(1H,brd,*J* = 5.2 Hz,H-6),3.51 (1H,m,H-3),0.67 (3H,s,H-18),1.02(3H,s,H-19),0.93 (3H,d,*J* = 8.1 Hz,H-21),0.83 (3H,d,*J* = 7.7 Hz,H-26),0.81 (3 H,d,*J* = 7.7 Hz,H-27),0.84 (3 H,t,*J* = 8.1 Hz,H-29);^{13}C NMR (125 MHz,$CDCl_3$):37.4 (t,C-1),31.8 (t,C-2),71.9 (d,C-3),42.5 (t,C-4),

140.9 (s,C-5),121.8 (d,C-6),32.1 (t,C-7),32.2 (d,C-8),50.3 (d,C-9),36.7 (s,C-10),21.2 (t,C-11),39.8 (t,C-12),42.5 (s,C-13),56.7 (d,C-14),24.2 (t,C-15),28.2 (t,C-16),56.3 (d,C-17),11.8 (q,C-18),19.2 (q,C-19),36.3 (d,C-20),18.9 (q,C-21),34.1 (t,C-22),26.4 (t,C-23),46.1(d,C-24),29.4 (d,C-25),19.9 (q,C-26),19.9 (q,C-27),23.3 (t,C-28),12.1 (q,C-29);EI-MS *m/z* (%):414 [M]$^+$(100),396 (59),381 (44),329 (51),303 (60),273 (40),255(40),231 (21),213 (41),173 (23),159 (41),145 (50),119 (42),105 (49),95 (55),69 (53),57 (67),43 (90)。以上数据与文献值相一致[10]。确定为:β-sitosterol。

化合物 10 3β,5α,9α-三羟基-麦角甾-7,22-二烯-6-酮,$C_{28}H_{44}O_4$,^{1}H NMR (500 MHz,C_5D_5N) δ:4.62 (1H, m, H-3),2.85 (1H, m, H-4α),5.93 (1H,br. s, H-7),2.96 (1H, br. t,8.7 Hz, H-14),0.66 (3H,s,H-18),1.15 (3H,s,H-19),1.06 (3H, d,6.4 Hz, H-21),5.21 (1H,dd,15.3 Hz,8.5,H-22),5.28 (1H,dd,15.4,7.8 Hz,H-23),0.87(3H, d,7.2 Hz,H-26),0.87 (3H,d,6.8 Hz,H-27),1.00 (3H,d,6.8 Hz,H-28),8.56 (1H,s,OH-5),6.32 (1H,br. s,OH-9);^{13}C NMR (125 MHz,C_5D_5N) δ:26.6(t,C-1),31.8 (t,C-2),66.8 (d,C-3),38.5 (t,C-4),79.9(s,C-5),199.3 (s,C-6),120.4 (d,C-7),164.3 (s,C-8),75.5 (s,C-9),42.4 (s,C-10),29.3 (t,C-11),35.6 (t,C-12),45.5 (s,C-13),52.3 (d,C-14),22.9 (t,C-15),28.4 (t,C-16),56.3 (d,C-17),12.6 (q,C-18),20.4 (q,C-19),40.8 (d,C-20),21.4 (q,C-21),136.4 (d,C-22),132.3 (d,C-23),43.4 (d,C-24),33.2 (d,C-25),19.8 (q,C-26),20.5 (q,C-27),17.9 (q,C-28)。上述数据与文献值相一致[11]。确定为:3β,5α,9α-三羟基-麦角甾-7,22-二烯-6-酮。

化合物 11 麦角甾-7,22-二烯-3-酮,$C_{28}H_{44}O$,无色针晶,^{1}H NMR (500 MHz ,$CDCl_3$) δ:0.58 (3H,s,H-18),0.82 (3H,d,6.4 Hz,H-26),0.84 (3H,d,6.6 Hz,H-27),0.92 (3H,d,6.9 Hz,H-28),1.02 (3H,s,H-19),1.03 (3H,d,6.7 Hz,H-21),5.17 (1H,dd,15.4,7.7 Hz,H-22),5.18 (1H,m,H-7),5.23 (1H,dd,15.2,7.0 Hz,H-23);^{13}C NMR (125 MHz ,$CDCl_3$) δ:38.6 (t,C-1),38.0 (t,C-2),212.1 (s,C-3),44.3 (t,C-4),42.9 (d,C-5),30.1 (t,C-6),117.1 (d,C-7),139.6 (s,C-8),48.9 (d,C-9),34.5 (s,C-10),21.8 (t,C-11),39.4 (t,C-12),43.3 (s,C-13),55.1 (d,C-14),22.8 (t,C-15),28.0 (t,C-16),55.8 (d,C-17),12.0 (q,C-18),12.3 (q,C-19),40.4 (d,C-20),21.0 (q,C-21),135.5 (d,C-22),131.8 (d,C-23),42.7 (d,C-24),33.0 (d,C-25),19.5 (q,C-26),19.8 (q,C-27),17.7 (q,C-28). EI-MS:396 (M^+,5),381 (2),353 (3),298 (13),271 (30),269 (87),244 (16),229 (25),213 (16)。上述数据与文献值相一致[12]。确定为:麦角甾-7,22-二烯-3-酮。

化合物 12 对羟基苯乙醇,无色结晶,^{1}H NMR (500 MHz,CD_3OD) δ:3.66 (2H,t,7.1 Hz,H-1),2.71 (2H,t,7.1 Hz,H-2),6.68 (2H,d,8.2 Hz,H-2′,H-6′),7.01 (2H, d, 8.2 Hz, H-3′, H-5′);^{13}C NMR (125 MHz,CD_3OD) δ:64.5 (t,C-1),39.5 (t,C-2),115.4 (s,C-1′),130.9 (d,C-2′,C-6′),116.0 (d,C-3′,C-5′),156.8 (s,C-4′);EI-MS *m/z* (%):138[M] (28),107(100),77(14)。以上数据与文献值相一致[13]。确定为:对羟基苯乙醇。

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