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# 黄药子的化学成分研究

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**摘　要**:综合利用各种色谱方法对黄药子的乙醇提取物进行分离,从中共得到23个化合物。通过波谱分析结合文献数据比对,将它们的结构分别鉴定为:2,7-二羟基-3,4-二甲氧基菲(**1**)、7-羟基-2,3,4-三甲氧基菲(**2**)、3,7-二羟基-2,4-二甲氧基-9,10-二氢菲(**3**)、3-羟基-2,4,7-三甲氧基-9,10-二氢菲(**4**)、2-羟基-3,4,7-三甲氧基-9,10-二氢菲(**5**)、7-羟基-2,3,4-三甲氧基-9,10-二氢菲(**6**)、7-羟基-2,4-二甲氧基-9,10-二氢菲(**7**)、2-羟基-4,7-二甲氧基-9,10-二氢菲(**8**)、2,7-二羟基-3,4-二甲氧基-9,10-二氢菲(**9**)、2′-羟基-3,4,5-三甲氧基联苄(**10**)、2′,3-二羟基-4,5-二甲氧基联苄(**11**)、3-羟基-4,5-二甲氧基联苄(**12**)、3,5-二羟基-4-甲氧基联苄(**13**)、pteryxin(**14**)、praeruptorin A(**15**)、山柰酚(**16**)、7,3′,4′-三羟基-3,5-二甲氧基黄酮(**17**)、黄独乙素(**18**)、diosbulbin G(**19**)、3-羟基-$\beta$-突厥酮(**20**)、(6*S*,9*S*)-吐叶醇(**21**)、(6*S*,9*R*)-吐叶醇(**22**)和blumenol B(**23**)。其中,化合物**4**~**8**、**10**、**12**~**15**、**20**~**23**共14个化合物均为首次从该植物中分离得到。

**关键词**:黄独;芪类;薯蓣属;角型香豆素;降倍半萜

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# Chemical Components from Rhizomes of *Dioscorea bulbiera*

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**Abstract**: Twenty-three compounds were isolated from the ethanol extracts of rhizomes of *Dioscorea bulbifera* by application of various chromatographic methods. Based on spectrum analyses in combination with literature data comparison, their structures were identified as 2,7-dihydroxy-3,4-dimethoxyphenanthrene (**1**), 7-hydroxy-2,3,4-trimethoxyphenanthrene (**2**), 3,7-dihydroxy-2,4-dimethoxy-9,10-dihydrophenanthrene (**3**), 3-hydroxy-2,4,7-trimethoxy-9,10-dihydrophenanthrene (**4**), 2-hydroxy-3,4,7-trimethoxy-9,10-dihydrophenanthrene (**5**), 7-hydroxy-2,3,4-trimethoxy-9,10-dihydrophenanthrene (**6**), 7-hydroxy-2,4-dimethoxy-9,10-dihydrophenanthrene (**7**), 2-hydroxy-4,7-dimethoxy-9,10-dihydrophenanthrene (**8**), 2,7-dihydroxy-3,4-dimethoxy-9,10-dihydrophenanthrene (**9**), 2′-hydroxy-3,4,5-trimethoxybibenzyl (**10**), 2′,3-dihydroxy-4,5-dimethoxybibenzyl (**11**), 3-hydroxy-4,5-dimethoxybibenzyl (**12**), 3,5-dihydroxy-4-methoxybibenzyl (**13**), pteryxin (**14**), praeruptorin A (**15**), kaempferol (**16**), 7,3′,4′-trihydroxy-3,5-methoxyflavone (**17**), diosbulbin B (**18**), diosbulbin G (**19**), 3-hydroxy-$\beta$-damascone (**20**), (6*S*,9*S*)-vomifoliol (**21**), (6*S*,9*R*)-vomifoliol (**22**), blumenol B (**23**), respectively. Among them, fourteen compounds including **4-8**, **10**, **12-15**, **20-23** were identified from *D. bulbifera* for the first time.

**Key words**: *Dioscorea bulbifera*; stilbenoids; *Dioscorea*; angular coumarins; norsesquiterpenoids

黄药子,为薯蓣科(Dioscoreaceae)薯蓣属(*Dioscorea*)缠绕草质藤本黄独(*Dioscorea bulbifera*)的块茎,具有消肿解毒,化痰散结,凉血止血等功效,可用于治疗瘿瘤,咳嗽痰喘,咳血,吐血,瘰疬,疮疡肿毒,毒蛇咬伤等症[1]。目前已报道黄药子的化学成分结构多样,主要包括甾类[2,3]、去甲克罗烷二萜内酯类[4-6]和多酚类[7,8]。现代药理试验研究表明黄药子在抗肿瘤、抗炎、抗菌、止痛、抗病毒等方面疗效确切,极具开发潜力[9,10]。然而,关于黄药子的毒副作用时有报道,尤其是肝损伤毒性,严重的限制其进一步的开发与利用[11,12]。有证据表明,其所含的二萜内酯类是导致肝损伤毒性的主要成分,具体作用机制与氧化应激密切相关[13-15]。作为黄药子中的代表性酚类,芪类化合物已表现出较好的抗肿瘤活

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性[16-18]。同时,该类成分是一类公认的天然抗氧化剂,已报道可通过抗氧化应激表现出保肝活性[19]。综上所述,萜类成分既是黄药子中的活性成分,亦可充当黄药子肝损伤毒性的潜在"减毒剂"。因此,为进一步明确黄药子物质基础,并为其"减毒"和质量控制奠定基础,本项目遂对黄药子的化学成分进行系统地研究,从中共分离得到 23 个化合物,其中 14 个为首次从该植物中分离得到。

## 1　仪器与试剂

核磁共振波谱仪(Bruker Ascend 400,TMS 为内标);ESI-MS 质谱仪(Waters Xevo TQ);柱层析硅胶和 $GF_{254}$薄层层析硅胶(青岛海洋化工有限公司);$C_{18}$反相柱层析硅胶(日本 YMC 公司);D101 大孔树脂购(天津海光化工有限公司);Sephadex LH-20 凝胶(GE Healthcare Bio-Sciences AB 公司);LC3000 制备型 HPLC 高效液相色谱仪(北京创新通恒科技有限公司);半制备型 HPLC 高效液相色谱仪(Perkin-Elmer 200);制备型 C-18 HPLC 色谱柱(YMC 公司,20 × 250 mm,10 μm);半制备型 C-18 HPLC 色谱柱(Welch 公司,10 × 250 mm,5 μm); HPLC 的检测波长均采用 208 nm;所有溶剂在使用前均经过蒸馏处理。

黄药子购于四川成都荷花池中药材市场。经中国科学院成都生物研究所高信芬研究员鉴定,所购药材为贵州产黄药子。样品标本(2014-08)存在成都生物研究所天然产物中心。

## 2　提取与分离

取干燥的黄药子粉末 100 kg,以 80 L 95% 乙醇在室温下浸提 3 次,每次 7 d。完毕,过滤,合并滤液,减压浓缩回收溶剂后得总浸膏。将总浸膏分散于 5 L 水中,依次以石油醚(5 L×3)、乙酸乙酯(5 L×3)和正丁醇(5 L×3)萃取,分别减压回收溶剂后得石油醚部位(P)580 g 和乙酸乙酯部位(E)900 g。

石油醚部位经硅胶柱层析(160 ~200 目),以石油醚/丙酮 (1∶0→10∶1)梯度洗脱得组分 P1-P5。组分 P4 经硅胶柱层析(160 ~200 目,石油醚∶丙酮 10∶1)进一步分成 5 个组分 P4A-P4E,其中组分 P4C 经半制备型 HPLC(甲醇/水 62∶38;流速 4 mL/min)分离得到化合物 **10**(5 mg,$t_R$ =31 min)。组分 P5 的甲醇溶解部分依次经制备型 HPLC(甲醇/水 68∶32;流速 15 mL/min)和半制备型 HPLC(甲醇/水 48∶52;流速 4 mL/min)分离得到化合物 **3**(5 mg,$t_R$ =44 min)和 **1**(4 mg,$t_R$ =51 min)。

乙酸乙酯部位经 D101 大孔树脂柱层析,以乙醇-水(0%、30%、60%、95%)梯度洗脱得 9 个组分 E1-E9。组分 E2 经凝胶 Sephadex LH-20 柱层析(氯仿/甲醇 1∶1)分离成 4 个亚组分 E2A-E2D。组分 E2A 依次经制备型 HPLC(甲醇/水 68∶32;流速 15 mL/min)和半制备型 HPLC(甲醇/水 62∶38;流速 3 mL/min)分离得到化合物 **14**(4 mg,$t_R$ =74 min)和 **15**(7 mg,$t_R$ =88 min)。组分 E2B 经制备型 HPLC(甲醇/水 69∶31;流速 15 mL/min)分成 3 个组分 E2B1($t_R$ =8 min)、E2B2($t_R$ =17 min)和 E2B3($t_R$ =24 min)。组分 E2B2 进一步经半制备型 HPLC(乙腈/水 48∶52;流速 4 mL/min)分离得到化合物 **12**(6 mg,$t_R$ =70 min)。组分 E2B3 经半制备型 HPLC(乙腈/水 48∶52;流速 4 mL/min)分离得到化合物 **4**(3 mg,$t_R$ =60 min)和 **5**(8 mg,$t_R$ =66 min)。组分 E2C 经半制备型 HPLC(甲醇/水 69∶31;流速 4 mL/min)分离得到化合物 **6**(4 mg,$t_R$ =11 min)。组分 E3 经凝胶 Sephadex LH-20 柱色谱(氯仿/甲醇 1∶1)和半制备型 HPLC(甲醇/水 63∶37;流速 4 mL/min)分离得到化合物 **2**(10 mg,$t_R$ =21 min)、**7**(4 mg,$t_R$ =27 min)和 **8**(7 mg,$t_R$ =29 min)。组分 E4 经凝胶 Sephadex LH-20 柱色谱(氯仿/甲醇 1∶1)分离得到 4 个亚组分 E4A-E4D。组分 E4A 依次经制备型 HPLC(甲醇/水 55∶45;流速 15 mL/min)、制备 TLC(氯仿/丙酮 10∶1)和半制备型 HPLC(甲醇/水 50∶50;流速 3 mL/min)分离得到化合物 **20**(7mg,$t_R$ =9 min)。组分 E5 经凝胶 Sephadex LH-20 柱色谱(氯仿/甲醇 1∶1)和半制备型 HPLC(乙腈/水 47∶53;流速 4 mL/min)分离得到化合物 **9**(5 mg,$t_R$ =16 min)、**11**(7 mg,$t_R$ =28 min)和 **13**(7 mg,$t_R$ =32 min)。组分 E6 的甲醇溶解部分经凝胶 Sephadex LH-20 柱色谱(氯仿/甲醇 1∶1)分离得到化合物 **16**(2 mg)和其它 5 个亚组分 E6A1-E6A5。其中,组分 E6A1 经 C18 反相硅胶柱色谱(甲醇/水 1∶1)分离得到亚组分 E6A1A 和 E6A1B。组分 E6A1A 经半制备型 HPLC(乙腈/水 17∶83;流速 4 mL/min)分离得到化合物 **23**(4 mg,$t_R$ =26 min)和亚组分 E6A1A2($t_R$ =28 min),后者进一步经半制备型 HPLC(甲醇/水 35∶65;流速 3 mL/min)分离得到化合物 **21**(6 mg,$t_R$ =29 min)和 **22**(10 mg,$t_R$ =30 min)。组分 E6A1B 经半制备型 HPLC(乙腈/水 27∶73;流速 4

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mL/min)分离得到化合物 **19**(6 mg,$t_R$ = 32 min)。组分 E6 的甲醇不溶部分经硅胶柱层析(氯仿/甲醇 15∶1)分离得到化合物 **18**(900 mg)。组分 E9 依次经凝胶 Sephadex LH-20 柱色谱(氯仿/甲醇 1∶1)和 MCI 柱色谱(甲醇/水 1∶1)分离得到化合物 **17**(80 mg)。

## 3 结构鉴定

**化合物 1** 红棕色粉末;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 9.36 (1H,d,$J$ = 9.1 Hz,H-5),7.53 (1H,d,$J$ = 8.9 Hz,H-10),7.49 (1H,d,$J$ = 8.9 Hz,H-9),7.20 (1H,d,$J$ = 2.8 Hz,H-8),7.17 (1H,dd,$J$ = 9.1,2.9 Hz,H-6),7.16 (1H,s,H-1),4.11 (3H,s,3-$OCH_3$),3.98 (3H,s,4-$OCH_3$);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 153.2 (C-7),150.7 (C-4),147.6 (C-2),140.9 (C-3),133.6 (C-8a),129.7 (C-10a),128.6 (C-10),127.4 (C-9),126.5 (C-5),124.4 (C-4b),118.9 (C-4a),116.5 (C-8),112.0 (C-6),108.3 (C-1),61.5 (3-$OCH_3$),59.9 (4-$OCH_3$);ESI-MS:$m/z$ 269.28 [M-H]$^-$。以上数据经与文献报道的数据进行比对后,确定该化合物为 2,7-二羟基-3,4-二甲氧基菲[20]。

**化合物 2** 红棕色油状物;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 9.40 (1H,d,$J$ = 9.1 Hz,H-5),7.57 (1H,d,$J$ = 8.8 Hz,H-10),7.51 (1H,d,$J$ = 8.8 Hz,H-9),7.20 (2H,m,H-6/8),7.07 (1H,s,H-1),4.04 (3H,s,3-$OCH_3$),4.01 (6H,s,2/4-$OCH_3$);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 153.3 (C-7),151.9 (C-2/4),143.0 (C-3),133.7 (C-8a),129.2 (C-10a),128.8 (C-10),127.4 (C-9),126.4 (C-5),124.3 (C-4b),119.3 (C-4a),116.7 (C-8),111.9 (C-6),105.5 (C-1),61.5 (3-$OCH_3$),60.4 (4-$OCH_3$),56.0 (2-$OCH_3$);ESI-MS:$m/z$ 283.12 [M-H]$^-$。以上数据经与文献报道的数据进行比对后,确定该化合物为 7-羟基-2,3,4-三甲氧基菲[18]。

**化合物 3** 淡黄色粉末;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 8.16 (1H,d,$J$ = 8.4 Hz,H-5),6.75 (1H,dd,$J$ = 8.4,2.7 Hz,H-6),6.72 (1H,d,$J$ = 2.7 Hz,H-8),6.57 (1H,s,H-1),3.91 (3H,s,4-$OCH_3$),3.70 (3H,s,2-$OCH_3$),2.72 (4H,m,H-9/10);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 154.2 (C-7),145.8 (C-2),144.9 (C-4),139.9 (C-8a),137.7 (C-3),129.8 (C-10a),128.4 (C-5),125.5 (C-4b),120.3 (C-4a),114.6 (C-6),113.6 (C-8),107.1 (C-1),60.2 (4-$OCH_3$),56.3 (2-$OCH_3$),30.3 (C-10),30.0 (C-9);ESI-MS:$m/z$ 271.27 [M-H]$^-$。以上数据经与文献报道的数据进行比对后,确定该化合物为 3,7-二羟基-2,4-二甲氧基-9,10-二氢菲[7]。

**化合物 4** 淡黄色油状物;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 8.21 (1H,d,$J$ = 8.7 Hz,H-5),6.83 (1H,dd,$J$ = 8.7,2.8 Hz,H-6),6.78 (1H,d,$J$ = 2.8 Hz,H-8),6.58 (1H,s,H-1),3.91 (3H,s,2-$OCH_3$),3.84 (3H,s,7-$OCH_3$),3.70 (3H,s,4-$OCH_3$),2.76 (2H,m,H-9),2.71 (2H,m,H-10);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 158.2 (C-7),145.8 (C-2),144.9 (C-4),139.6 (C-8a),137.7 (C-3),129.8 (C-10a),128.2 (C-5),125.4 (C-4b),120.4 (C-4a),113.4 (C-8),111.9 (C-6),107.1 (C-1),60.2 (4-$OCH_3$),56.3 (2-$OCH_3$),55.3 (7-$OCH_3$),30.5 (C-10),30.1 (C-9);ESI-MS:$m/z$ 285.12 [M-H]$^-$。以上数据经与文献报道的数据进行比对后,确定该化合物为 3-羟基-2,4,7-三甲氧基-9,10-二氢菲[17]。

**化合物 5** 淡黄色油状物;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 8.18 (1H,d,$J$ = 8.7 Hz,H-5),6.81 (1H,dd,$J$ = 8.7,2.9 Hz,H-6),6.77 (1H,d,$J$ = 2.8 Hz,H-8),6.63 (1H,s,H-1),3.97 (3H,s,3-$OCH_3$),3.84 (3H,s,7-$OCH_3$),3.75 (3H,s,4-$OCH_3$),2.74 (2H,m,H-9),2.69 (2H,m,H-10);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 157.9 (C-7),150.5 (C-4),147.6 (C-2),139.4 (C-8a),139.0 (C-3),134.9 (C-10a),128.3 (C-5),125.4 (C-4b),120.3 (C-4a),113.1 (C-8),111.7 (C-6),110.1 (C-1),61.1 (3-$OCH_3$),60.0 (4-$OCH_3$),55.2 (7-$OCH_3$),30.2 (C-10),30.0 (C-9);ESI-MS:$m/z$ 285.27 [M-H]$^-$。以上数据经与文献报道的数据进行比对后,确定该化合物为 2-羟基-3,4,7-三甲氧基-9,10-二氢菲[21]。

**化合物 6** 淡黄色油状物;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 8.17 (1H,d,$J$ = 8.5 Hz,H-5),6.74 (1H,dd,$J$ = 8.5,2.8 Hz,H-6),6.77 (1H,d,$J$ = 2.6 Hz,H-8),6.58 (1H,s,H-1),3.91 (3H,s,3-$OCH_3$),3.88 (3H,s,2-$OCH_3$),3.77 (3H,s,4-$OCH_3$),2.74 (2H,m,H-9),2.69 (2H,m,H-10);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 154.2 (C-7),151.7 (C-3),151.7 (C-2),141.6 (C-4),139.8 (C-8a),134.2 (C-10a),128.8 (C-5),125.6 (C-4b),121.0 (C-4a),

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114.6 (C-8),113.4 (C-6),107.5 (C-1),61.2 (3-$OCH_3$),60.6 (4-$OCH_3$),56.1 (2-$OCH_3$),30.4 (C-10),30.2 (C-9);ESI-MS:*m/z* 285.09 [M-H]$^-$。以上数据经与文献报道的数据进行比对后,确定该化合物为7-羟基-2,3,4-三甲氧基-9,10-二氢菲[16]。

**化合物7** 淡黄色粉末;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 8.12 (1H,d,*J*=8.4 Hz,H-5),6.71 (2H,m,H-6/8),6.46 (1H,d,*J*=2.5 Hz,H-1),6.42 (1H,d,*J*=2.5 Hz,H-3),3.87 (3H,s,4-$OCH_3$),3.84 (3H,s,2-$OCH_3$),2.73 (4H,m,H-9/10);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 158.8 (C-7),157.8 (C-4),153.8 (C-2),140.4 (C-10a),139.9 (C-8a),129.3 (C-5),125.8 (C-4b),116.7 (C-4a),114.4 (C-8),112.9 (C-6),105.0 (C-1),97.7 (C-3),55.7 (4-$OCH_3$),55.5 (2-$OCH_3$),30.9 (C-10),30.1 (C-9);ESI-MS:*m/z* 255.21 [M-H]$^-$。以上数据经与文献报道的数据进行比对后,确定该化合物为7-羟基-2,4-二甲氧基-9,10-二氢菲[22]。

**化合物8** 淡黄色粉末;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 8.17 (1H,d,*J*=8.7 Hz,H-5),6.81 (1H,dd,*J*=8.7,2.9 Hz,H-6),6.78 (1H,d,*J*=2.8 Hz,H-8),6.41 (1H,d,*J*=2.4 Hz,H-1),6.34 (1H,d,*J*=2.4 Hz,H-3),3.87 (3H,s,4-$OCH_3$),3.84 (3H,s,7-$OCH_3$),2.72 (4H,m,H-9/10);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 157.9 (C-4),157.6 (C-7),154.7 (C-2),141.2 (C-10a),140.0 (C-8a),129.0 (C-5),125.7 (C-4b),116.6 (C-4a),113.3 (C-8),111.3 (C-6),107.4 (C-1),98.2 (C-3),55.7 (4-$OCH_3$),55.4 (7-$OCH_3$),30.6 (C-10),30.2 (C-9);ESI-MS:*m/z* 255.11 [M-H]$^-$。以上数据经与文献报道的数据进行比对后,确定该化合物为2-羟基-4,7-二甲氧基-9,10-二氢菲[17]。

**化合物9** 黄色粉末;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 8.13 (1H,d,*J*=8.4 Hz,H-5),6.73 (1H,dd,*J*=8.5,2.8 Hz,H-6),6.70 (1H,d,*J*=2.7 Hz,H-8),6.62 (1H,s,H-1),3.97 (3H,s,3-$OCH_3$),3.74 (3H,s,4-$OCH_3$),2.69 (4H,m,H-9/10);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 154.0 (C-7),150.6 (C-4),147.6 (C-2),139.9 (C-8a),139.1 (C-3),135.0 (C-10a),128.9 (C-5),125.7 (C-4b),120.4 (C-4a),114.6 (C-8),113.4 (C-6),110.2 (C-1),61.3 (3-$OCH_3$),60.2 (4-$OCH_3$),30.1 (C-9/10);ESI-MS:*m/z* 271.29 [M-H]$^-$。以上数据经与文献报道的数据进行比对后,确定该化合物为2,7-二羟基-3,4-二甲氧基-9,10-二氢菲[20]。

**化合物10** 淡黄色油状物;$^1$H NMR (400 MHz,$CDCl_3$):$\delta$ 7.08 (2H,m,H-4′/6′),6.84 (1H,t,*J*=7.4 Hz,H-5′),6.76 (1H,d,*J*=8.1 Hz,H-6′),6.38 (2H,s,H-2/6),3.82 (3H,s,4-$OCH_3$),3.81 (6H,s,3/5-$OCH_3$),2.87 (4H,m,H-7/7′);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 153.9 (C-2′),153.2 (C-3/5),138.0 (C-1),136.3 (C-4),130.5 (C-6′),128.0 (C-1′),127.4 (C-4′),120.8 (C-5′),115.5 (C-3′),105.5 (C-2/6),61.0 (4-$OCH_3$),56.2 (3/5-$OCH_3$),36.7 (C-7′),32.5 (C-7);ESI-MS:*m/z* 287.19 [M-H]$^-$。以上数据经与文献报道的数据进行比对后,确定该化合物为2′-羟基-3,4,5-三甲氧基联苄[7,23]。

**化合物11** 淡黄色油状物;$^1$H NMR (400 MHz,$CDCl_3$):$\delta$ 7.09 (2H,t,*J*=7.4 Hz,H-4′/6′),6.86 (1H,td,*J*=7.4,1.2 Hz,H-5′),6.75 (1H,d,*J*=7.7 Hz,H-3′),6.50 (1H,d,*J*=1.8 Hz,H-2),6.26 (1H,d,*J*=1.8 Hz,H-6),3.87 (3H,s,4-$OCH_3$),3.80 (3H,s,5-$OCH_3$),2.89 (2H,m,H-7),2.84 (2H,m,H-7′);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 153.7 (C-2′),152.3 (C-5),149.2 (C-3),138.5 (C-1),133.9 (C-4),130.5 (C-6′),127.9 (C-1′),127.5 (C-4′),121.0 (C-5′),115.5 (C-3′),108.0 (C-2),104.6 (C-6),61.1 (4-$OCH_3$),55.9 (5-$OCH_3$),36.4 (C-7′),32.4 (C-7);ESI-MS:*m/z* 273.13 [M-H]$^-$。以上数据经与文献报道的数据进行比对后,确定该化合物为2′,3-二羟基-4,5-二甲氧基联苄[7]。

**化合物12** 淡黄色油状物 $^1$H NMR (400 MHz,$CDCl_3$):$\delta$ 7.29 (2H,m,H-3′/5′),7.19 (3H,m,H-2′/4′/6′),6.47 (1H,d,*J*=1.7 Hz,H-2),6.24 (1H,d,*J*=1.7 Hz,H-6),3.87 (3H,s,4-$OCH_3$),3.81 (3H,s,5-$OCH_3$),2.90 (2H,m,H-7′),2.82 (2H,m,H-7);$^{13}$C NMR (100 MHz,$CDCl_3$):$\delta$ 152.22 (C-5),149.24 (C-3),141.82 (C-1′),138.25 (C-1),133.84 (C-4),128.60 (C-3′/5′),128.48 (C-2′/6′),126.08 (C-4′),108.01 (C-2),104.56 (C-6),61.11 (4-$OCH_3$),55.92 (5-$OCH_3$),38.16 (C-7′),37.96 (C-7);ESI-MS:*m/z* 257.11

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[M-H]⁻。以上数据经与文献报道的数据进行比对后,确定该化合物为3-羟基-4,5-二甲氧基联苄[24]。

**化合物13** 淡黄色油状物;$^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.29 (2H, t, $J$ = 7.7 Hz, H-3′/5′), 7.19 (3H, t, $J$ = 7.7 Hz, H-2′/4′/6′), 6.35 (2H, br s, H-2/6), 3.85 (3H, s, 4-$OCH_3$), 2.87 (2H, m, H-7′), 2.77 (2H, m, H-7); $^1$H NMR (400 MHz, MeOH-$d_4$): $\delta$ 7.23 (2H, m, H-3′/5′), 7.19 (3H, m, H-2′/4′/6′), 6.20 (2H, br s, H-2/6), 3.76 (3H, s, 4-$OCH_3$), 2.83 (2H, m, H-7′), 2.69 (2H, m, H-7); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 148.7 (C-3/5), 141.8 (C-1′), 139.1 (C-1), 132.8 (C-4), 128.5 (C-3′/5′), 128.5 (C-2′/6′), 126.1 (C-4′), 108.2 (C-2/6), 61.4 (4-$OCH_3$), 37.7 (C-7/7′); ESI-MS: $m/z$ 243.19 [M-H]⁻。以上数据经与文献报道的数据进行比对后,确定该化合物为3,5-二羟基-4-甲氧基联苄[25]。

**化合物14** 淡黄色油状物;$^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.59 (1H, d, $J$ = 9.5 Hz, H-4), 7.36 (1H, d, $J$ = 8.6 Hz, H-5), 6.80 (1H, d, $J$ = 8.6 Hz, H-6), 6.63 (1H, d, $J$ = 4.9 Hz, H-4′), 6.22 (1H, d, $J$ = 9.5 Hz, H-3), 6.03 (1H, qd, $J$ = 7.1, 1.5 Hz, H-3″), 5.35 (1H, d, $J$ = 4.9 Hz, H-3′), 2.09 (3H, s, 3′-$OCOCH_3$), 2.00 (3H, dd, $J$ = 7.2, 1.5 Hz, H-4″), 1.86 (3H, br t, $J$ = 1.5 Hz, H-5″), 1.46 (3H, s, H-6′), 1.43 (3H, s, H-5′); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 170.0 (3′-$\underline{OCOCH_3}$), 167.1 (C-1″), 159.9 (C-2), 156.8 (C-7), 154.2 (C-9), 143.3 (C-4), 138.0 (C-3″), 129.4 (C-5), 127.6 (C-2″), 114.6 (C-6), 113.5 (C-3), 112.7 (C-10), 107.5 (C-8), 77.4 (C-2′), 70.7 (C-3′), 60.3 (C-4′), 25.5 (C-5′), 22.4 (C-6′), 20.9 (3′-$OCO\underline{CH_3}$), 20.5 (C-5″), 15.7 (C-4″); ESI-MS: $m/z$ 409.17 [M + Na]⁺。以上数据经与文献报道的数据进行比对后,确定该化合物为 pteryxin[26,27]。

**化合物15** 淡黄色油状物;$^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.62 (1H, d, $J$ = 9.5 Hz, H-4), 7.37 (1H, d, $J$ = 8.6 Hz, H-5), 6.82 (1H, d, $J$ = 8.6 Hz, H-6), 6.61 (1H, d, $J$ = 4.8 Hz, H-4′), 6.25 (1H, d, $J$ = 9.5 Hz, H-3), 6.15 (1H, qd, $J$ = 7.3, 1.6 Hz, H-3″), 5.42 (1H, d, $J$ = 4.8 Hz, H-3′), 2.12 (3H, s, 4′-$OCOCH_3$), 1.97 (3H, dd, $J$ = 7.2, 1.6 Hz, H-4″), 1.88 (3H, t, $J$ = 1.7 Hz, H-5″), 1.49 (3H, s, H-6′), 1.45 (3H, s, H-5′). $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 169.9 (4′-$\underline{OCOCH_3}$), 166.6 (C-1″), 160.0 (C-2), 156.8 (C-7), 154.1 (C-9), 143.5 (C-4), 139.9 (C-3″), 129.3 (C-5), 127.1 (C-2″), 114.5 (C-6), 113.3 (C-3), 112.6 (C-10), 107.1 (C-8), 77.8 (C-2′), 69.9 (C-3′), 61.1 (C-4′), 25.0 (C-5′), 23.1 (C-6′), 20.8 (4′-$OCOCH_3$), 20.6 (C-5″), 15.9 (C-4″); ESI-MS: $m/z$ 409.23 [M + Na]⁺。以上数据经与文献报道的数据进行比对后,确定该化合物为 praeruptorin A[26,28]。

**化合物16** 黄色固体;$^1$H NMR (400 MHz, Acetone-$d_6$): $\delta$ 12.18 (1H, s, 5-OH), 8.14 (2H, d, $J$ = 8.9 Hz, H-2′/6′), 7.01 (2H, d, $J$ = 8.9 Hz, H-3′/5′), 6.52 (1H, d, $J$ = 1.9 Hz, H-8), 6.26 (1H, d, $J$ = 1.9 Hz, H-6); ESI-MS: 285.14 [M-H]⁻。以上数据经与文献报道的数据进行比对后,确定该化合物为山柰酚[29]。

**化合物17** 黄色粉末;$^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 7.49 (1H, d, $J$ = 2.2 Hz, H-2′), 7.37 (1H, dd, $J$ = 8.5, 2.2 Hz, H-6′), 6.87 (1H, d, $J$ = 8.5 Hz, H-5′), 6.42 (1H, d, $J$ = 2.0 Hz, H-8), 6.33 (1H, d, $J$ = 2.1 Hz, H-6), 3.79 (3H, s, 5-$OCH_3$), 3.69 (3H, s, 3-$OCH_3$); $^{13}$C NMR (100 MHz, DMSO-$d_6$): $\delta$ 172.3 (C-4), 162.6 (C-7), 160.8 (C-5), 158.1 (C-9), 152.0 (C-2), 148.0 (C-4′), 145.2 (C-3′), 139.8 (C-3), 121.3 (C-1′), 120.1 (C-6′), 115.8 (C-2′), 115.2 (C-5′), 96.2 (C-6), 94.8 (C-8), 59.3 (5-$OCH_3$), 56.0 (3-$OCH_3$); ESI-MS: 329.27 [M-H]⁻。以上数据经与文献报道的数据进行比对后,确定该化合物为7,3′,4′-三羟基-3,5-二甲氧基黄酮[30]。

**化合物18** 黄白色粉末;$^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.57 (1H, d, $J$ = 1.4 Hz, H-16), 7.40 (1H, d, $J$ = 1.8 Hz, H-15), 6.94 (1H, d, $J$ = 1.1 Hz, H-14), 5.30 (1H, dd, $J$ = 11.0, 5.4 Hz, H-12), 4.88 (1H, m, H-2), 4.71 (1H, d, $J$ = 5.6 Hz, H-6), 2.76 (1H, d, $J$ = 5.3 Hz, H-4), 2.61 (1H, dd, $J$ = 11.6, 5.7 Hz, H-7a), 2.56 (1H, m, H-3a), 1.97 (1H, dd, $J$ = 12.2, 5.5 Hz, H-1a), 1.93 (1H, d, $J$ = 11.4 Hz, H-3b), 1.80 (1H, d, $J$ = 11.8 Hz, H-11b), 1.61 (1H, d, $J$ = 13.4 Hz, H-1b), 1.25 (3H, s, H-19); $^{13}$C NMR (100 MHz, $CDCl_3$): $\delta$ 176.4 (C-17), 175.3 (C-18), 143.6 (C-15), 141.5 (C-16), 125.2 (C-13), 110.2

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(C-14),89.4 (C-8),77.4 (C-6),76.5 (C-2),75.4 (C-12),45.9 (C-9),43.0 (C-4),42.3 (C-5),42.0 (C-11),39.6 (C-3),38.7 (C-10),37.5 (C-7),29.8 (C-1),16.8 (C-19);ESI-MS:*m/z* 343.17 [M-H]$^-$,367.23 [M + Na]$^+$。以上数据经与文献报道的数据进行比对后,确定该化合物为黄独乙素[31]。

**化合物 19** 黄白色粉末;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 7.48 (1H,br. s,H-16),7.44 (1H,br s,H-15),6.44 (1H,br s,H-14),5.39 (1H,dd,$J$ = 11.2, 6.1 Hz,H-12),4.56 (1H,br s,H-6),4.29 (1H,br s,H-2),2.93 (1H,d,$J$ = 12.3,4.6 Hz,H-8),2.84 (1H,t,$J$ = 5.8 Hz,H-4),2.56 (1H,ddd,$J$ = 16.3, 4.7,2.1 Hz H-7a),1.25 (1H,td,$J$ = 13.3,2.3,H-1b),0.95 (3H,s,H-19);$^1$H NMR (400 MHz,Pyridine-$d_5$):$\delta$ 7.78 (1H,br. s,H-16),7.66 (1H,d,$J$ = 1.9 Hz,H-15),6.64 (1H,br s,H-14),5.40 (1H,dd,$J$ = 11.5,5.8 Hz,H-12),4.48 (1H,br s,H-6),4.29 (1H,br s,H-2),3.08 (1H,d,$J$ = 12.6,4.5 Hz,H-8),2.91 (1H,dt,$J$ = 7.6,3.9 Hz,H-4),2.55 (2H,m,H-7),2.45 (1H,t,$J$ = 12.1 Hz,H-3a),1.24 (1H,t,$J$ = 12.5 Hz,H-1b),0.90 (3H,s,H-19);$^{13}$C NMR (100 MHz, $CDCl_3$):$\delta$ 178.6 (C-17),173.7 (C-18),143.9 (C-15),139.8 (C-16),124.3 (C-13),108.7 (C-14),76.6 (C-6),70.4 (C-12),65.8 (C-2),42.5 (C-8),42.2 (C-10),41.6 (C-11),40.1 (C-5),37.0 (C-4),34.9 (C-9),30.7 (C-3),30.0 (C-1),24.0 (C-7),16.8 (C-19);ESI-MS:*m/z* 343.17 [M-H]$^-$,367.23 [M + Na]$^+$。以上数据经与文献报道的数据进行比对后,确定该化合物为 Diosbulbin G[32]。

**化合物 20** 无色油状物;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 6.72 (1H, dq, $J$ = 13.8, 6.9 Hz, H-9), 6.14 (1H, d, $J$ = 13.8 Hz, H-8), 4.07 (1H, m, H-3), 2.35 (1H, dd, $J$ = 16.8, 5.7 Hz, H-4a), 2.01 (1H, dd, $J$ = 16.9, 9.5 Hz, H-4b), 1.92 (1H, dd, $J$ = 6.9, 1.0 Hz, H-10), 1.53 (3H, s, 5-$CH_3$), 1.47 (1H, d, $J$ = 11.9 Hz, H-2b), 1.14 (3H, s, 1-$CH_3$), 0.98 (3H, s, 1-$CH_3$);$^{13}$C NMR (100 MHz, $CDCl_3$):$\delta$ 201.8 (C-7), 146.4 (C-9), 140.1 (C-5), 134.6 (C-8), 128.2 (C-6), 65.0 (C-3), 48.0 (C-2), 41.0 (C-4), 36.5 (C-1), 29.8 (1-$CH_3$), 29.2 (2-$CH_3$), 21.2 (5-$CH_3$), 18.5 (C-10); ESI-MS: *m/z* 209.21 [M + H]$^+$。以上数据经与文献报道的数据进行比对后,确定该化合物为 3-羟基-$\beta$-突厥酮[33]。

**化合物 21** 无色油状物;$[\alpha]_D^{20}$ +204 (*c* 0.29, MeOH);$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 5.89 (1H, s, H-4), 5.86 (1H, dd, $J$ = 15.7, 5.2 Hz, H-8), 5.77 (1H, d, $J$ = 15.7 Hz, H-7), 4.40 (1H, m, H-9), 2.44 (1H, d, $J$ = 17.1 Hz, H-2a), 2.23 (1H, d, $J$ = 17.1 Hz, H-2b), 1.89 (3H, s, 5-$CH_3$), 1.29 (3H, d, $J$ = 6.4 Hz, H-10), 1.07 (3H, s, 1-$CH_3$), 0.99 (3H, s, 1-$CH_3$);$^{13}$C NMR (100 MHz, $CDCl_3$):$\delta$ 198.2 (C-3), 163.1 (C-5), 135.9 (C-8), 129.1 (C-7), 127.0 (C-4), 79.2 (C-6), 68.1 (C-9), 49.9 (C-2), 41.3 (C-1), 24.2 (1-$CH_3$), 23.9 (C-10), 23.0 (1-$CH_3$), 19.1 (5-$CH_3$); ESI-MS: *m/z* 225.07 [M + H]$^+$。以上数据经与文献报道的数据进行比对后,确定该化合物为(6*S*,9*S*)-吐叶醇[34]。

**化合物 22** 无色油状物;$[\alpha]_D^{20}$ +220 (*c* 0.11, MeOH);$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 5.87 (1H, s, H-4), 5.82 (1H, dd, $J$ = 15.6, 5.1 Hz, H-8), 5.75 (1H, d, $J$ = 15.7 Hz, H-7), 4.37 (1H, p, $J$ = 6.2 Hz, H-9), 2.41 (1H, d, $J$ = 17.2 Hz, H-2a), 2.20 (1H, d, $J$ = 17.0 Hz, H-2b), 1.88 (3H, s, 5-$CH_3$), 1.27 (3H, d, $J$ = 6.4 Hz, H-10), 1.05 (3H, s, 1-$CH_3$), 0.99 (3H, s, 1-$CH_3$);$^{13}$C NMR (100 MHz, $CDCl_3$):$\delta$ 198.5 (C-3), 163.6 (C-5), 135.8 (C-8), 129.1 (C-7), 126.8 (C-4), 79.1 (C-6), 68.1 (C-9), 49.8 (C-2), 41.3 (C-1), 24.1 (1-$CH_3$), 23.8 (C-10), 23.1 (1-$CH_3$), 19.1 (5-$CH_3$); ESI-MS: *m/z* 225.17 [M + H]$^+$。以上数据经与文献报道的数据进行比对后,确定该化合物为(6*S*,9*R*)-吐叶醇[34]。

**化合物 23** 无色油状物;$^1$H NMR (400 MHz, $CDCl_3$):$\delta$ 5.84 (1H, s, H-4), 3.82 (1H, m, H-9), 2.52 (1H, d, $J$ = 18.0 Hz, H-2a), 2.22 (1H, d, $J$ = 18.0 Hz, H-2b), 2.02 (3H, s, 5-$CH_3$), 1.91 (2H, m, H-7), 1.53 (2H, m, H-8), 1.23 (1H, d, $J$ = 6.2 Hz, H-10), 1.09 (3H, s, 1-$CH_3$), 1.05 (3H, s, 1-$CH_3$);$^{13}$C NMR (100 MHz, $CDCl_3$):$\delta$ 198.1 (C-3), 167.8 (C-5), 126.2 (C-4), 77.9 (C-6), 68.9 (C-9), 50.2 (C-2), 41.9 (C-1), 33.7 (C-7), 33.5 (C-8), 24.1 (1-$CH_3$), 24.0 (1/9-$CH_3$), 21.1 (5-$CH_3$); ESI-MS: *m/z* 227.34 [M + H]$^+$。以上数据经与文献报道的数据进行比对后,确定该化合物为 Blumenol B[35]。

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