

应阳性,提示含有酚羟基。该化合物的<sup>1</sup>H-NMR谱中,9.97(1H,s)为醛基氢特征信号,其碳信号为190.7(CH)。芳香质子区存在一组互为邻位偶合的氢信号:7.74(2H,d,J=8.4 Hz)、6.91(2H,d,J=8.4 Hz)。<sup>13</sup>C-NMR谱中,存在1个sp<sup>2</sup>杂化连氧碳信号(163.2),推测醛基的对位被羟基取代,故将化合物B-14鉴定为4-羟基苯甲醛(4-hydroxybenzaldehyde),经与文献对照基本一致<sup>[10]</sup>。

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### 福建产金线莲中黄酮苷成分的研究

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**摘要:**目的 对兰科开唇兰属植物福建产金线莲 *Ancecotichilus roxburghii* 提取物中的正丁醇萃取部分进行分离纯化和结构鉴定。方法 利用硅胶柱色谱、凝胶 LH-20 和反相高效液相制备等方法进行分离纯化,使用快原子轰击质谱、核磁共振等现代波谱技术确定其结构。结果 从福建产金线莲 95%乙醇渗漉提取物正丁醇部分得到 5 个黄酮苷类化合物,其结构为:槲皮素-7-O-β-D-葡萄糖苷(I)、槲皮素-3-O-β-D-芸香糖苷(II)、异鼠李素-3,4'-O-β-D-二葡萄糖苷(III)、异鼠李素-3,7-O-β-D-二葡萄糖苷(IV)、异鼠李素-7-O-β-D-二葡萄糖苷(V)。结论 5 种化合物均为首次从该植物中分得,除化合物 II 外均为首次从兰科植物中分离得到。从福建产金线莲中得到大量黄酮类化合物,推测黄酮为该植物中主要活性成分。

**关键词:**兰科;开唇兰属;金线莲;黄酮苷

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### Isolation and structural elucidation of flavonoids from *Ancecotichilus roxburghii*

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**Abstract; Objective** To isolate and identify the chemical constituents in the polar part of the *Ancecotichilus roxburghii*. **Methods** The flavonoids were isolated by column chromatography and their structures were elucidated through spectral analyses such as FAB-MS and NMR. **Results** Five flavonoids were isolated and identified as quercetin 7-O-β-D-glucoside (I), quercetin 3-O-β-D-rutinoside (II), isorham-

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netin-3, 4'-O- $\beta$ -D-diglucoiside (III), isorhamnetin-3, 7-O- $\beta$ -D-diglucoiside (IV), and isorhamnetin-7-O- $\beta$ -D-diglucoiside (V). **Conclusion** All the compounds are obtained from *A. roxburghii* for the first time.

**Key words:** Orchidaceae; *Anoectochilus* Bl.; *Anoectochilus roxburghii* (Wall.) Lindl.; flavonoid

福建产金线莲 *Anoectochilus roxburghii* (Wall.) Lindl. 为兰科开唇兰属珍稀药用植物,又名金线兰、花叶开唇兰等。该植物主要分布于中国南部福建等省,日本、斯里兰卡、印度和尼泊尔等国也有零星分布<sup>[1]</sup>。民间有“药王”、“金草”之美誉,其味甘、微苦,性平微寒,具有清热凉血、祛风除湿、强心、固肾、利尿、平肝、降血压、解毒、止痛、镇咳等功效,民间多用于治疗咯血、支气管炎、肾炎、膀胱炎、糖尿病、乳糜尿、血尿、风湿性关节炎、小儿急惊风、毒蛇咬伤等,近年来主要用于治疗高血压、糖尿病、心脏病、肺炎、急性慢性肝炎、肾炎等症<sup>[2]</sup>。对该植物的研究主要集中在文献考证、人工栽培、组培、商品调查等方面<sup>[3-8]</sup>,对其化学成分研究未见报道,药理活性的报道很少,为寻找其活性物质,为建立质量标准等奠定基础,对福建金线莲的化学成分进行了系统的分离。从该植物正丁醇部分分得5个化合物,其结构分别为:槲皮素-7-O- $\beta$ -D-葡萄糖苷(I)、槲皮素-3-O- $\beta$ -D-芸香糖苷(II)、异鼠李素-3,4'-O- $\beta$ -D-二葡萄糖苷(III)、异鼠李素-3,7-O- $\beta$ -D-二葡萄糖苷(IV)、异鼠李素-7-O- $\beta$ -D-二葡萄糖苷(V)。5种化合物均为首次从该植物中分得,除化合物III外均为首次从兰科植物中分得。

### 1 仪器与材料

X-4 数字显示显微熔点测定仪(温度计未校正),Peking-Elmer 983G 型红外光谱仪(溴化钾压片),Bruker AM-500 型核磁共振仪,内标为 TMS(本实验所测<sup>1</sup>H-NMR和<sup>13</sup>C-NMR均为 500 MHz),Autospec-Ultima ETOF 质谱仪,BUCHI R-3000 型旋转蒸发仪,柱色谱用硅胶(青岛海洋化工厂),聚酰胺粉(浙江台州市路桥四甲生化塑料厂),AB-8 大孔树脂(天津南开大学化工厂),Sephadex LH-20 (Pharmacia 公司),GF254 和 HF254 高效薄层板(烟台汇友硅胶开发有限公司)。实验所用试剂均为分析纯。金线莲购于福建省,由中国医学科学院药用植物研究所郭顺星研究员鉴定为 *A. roxburghii* (Wall.) Lindl.。

### 2 提取与分离

金线莲干全草 5.7 kg,切碎,95%乙醇渗漉法提取,回收溶剂,得 580 g 浸膏。所得浸膏按经典分离方法进行萃取粗分,分成正己烷、氯仿、醋酸乙

酯、正丁醇、水 5 部分。正丁醇部分 100 g,经大孔树脂色谱,50%乙醇洗脱部分得 37 g 浸膏,以氯仿-甲醇-水梯度洗脱,经反复硅胶柱、聚酰胺柱色谱和 Sephadex LH-20 纯化,分别得化合物 I (3.5 mg)、II (15 mg)、III (10 mg)、IV (20 mg)、V (15 mg)。

### 3 结构鉴定

化合物 I:黄色结晶性粉末,mp 173~175 °C (甲醇),HCl-Mg 反应呈阳性,Molish 反应呈阳性,分子式:C<sub>21</sub>H<sub>20</sub>O<sub>12</sub>。FAB-MS *m/z*:467.2(M<sup>+</sup>+H+D),304(M<sup>+</sup>D-162),316(A<sub>1</sub>+162)。进行薄层酸水解,与标准糖对照,表明含有葡萄糖。UV  $\lambda_{\max}^{\text{MeOH}}$  nm:365,252。<sup>1</sup>H-NMR(DMSO-d<sub>6</sub>) $\delta$ :7.76(1H,d,J=1.5 Hz,H-2'),6.89(1H,d,J=8.5 Hz,H-5'),7.66(1H,dd,J=1.5,8.5 Hz,H-6'),6.46(1H,d,J=2 Hz,H-6);6.74(1H,d,J=2 Hz,H-8),5.07(1H,d,J=6 Hz,H-1'')。 <sup>13</sup>C-NMR(DMSO-d<sub>6</sub>) $\delta$ :147.78(C-2),136.19(C-3),176.31(C-4),160.94(C-5),99.03(C-6),163.25(C-7),94.28(C-8),156.50(C-9),105.09(C-10),122.81(C-1'),116.60(C-2'),147.58(C-3'),145.05(C-4'),114.50(C-5'),120.74(C-6'),100.49(C-1''),73.59(C-2''),76.45(C-3''),69.69(C-4''),77.19(C-5''),61.30(C-6'')。综合以上数据,推测该化合物结构为 3',4',5-三羟基黄酮醇-7-O- $\beta$ -D-葡萄糖苷,即 quercimeritroside。经与文献核对<sup>[9,10]</sup>,各种数据基本符合。

化合物 II:黄色固体,mp 158~160 °C(甲醇),HCl-Mg 反应呈阳性,Molish 反应呈阳性,分子式:C<sub>27</sub>H<sub>30</sub>O<sub>16</sub>,进行薄层酸水解,与标准糖对照,表明含有葡萄糖和鼠李糖。UV  $\lambda_{\max}$  nm:365,257,205(sh)。FAB-MS *m/z*:611.2(M<sup>+</sup>+H),303.1(M<sup>+</sup>+H162-146),137(B<sub>1</sub>),154(A<sub>1</sub>+H)。<sup>1</sup>H-NMR(DMSO-d<sub>6</sub>) $\delta$ :7.68(1H,d,J=2 Hz,H-2'),7.65(1H,dd,J=2,8.5 Hz,H-6'),6.89(1H,d,J=8.5 Hz,H-5'),6.42(1H,d,J=2 Hz,H-8),6.23(1H,d,J=2 Hz,H-6),5.13(1H,d,J=7.5 Hz,-Glc H-1),4.54(1H,s,-Rha H-1),1.40(2H,d,J=6.0 Hz,-Rha H-6)。<sup>13</sup>C-NMR(DMSO-d<sub>6</sub>) $\delta$ : $\delta$  159.36(C-2),135.61(C-3),179.45(C-4),163.01(C-5),99.95(C-6),166.05(C-7),94.86(C-8),158.54(C-

9), 105.45 (C-10), 123.55 (C-1'), 117.68 (C-2'), 149.82 (C-3'), 145.86 (C-4'), 116.06 (C-5'), 123.13 (C-6'); -Glc: 104.68 (C-1), 75.73 (C-2), 78.18 (C-3), 71.40 (C-4), 77.24 (C-5), 68.54 (C-6); -Rha: 102.42 (C-1), 72.23 (C-2), 72.11 (C-3), 73.93 (C-4), 69.72 (C-5), 17.88 (C-6)。综合以上数据, 推定该化合物结构为 3', 4', 5, 7-四羟基黄酮醇-3-O-β-D-芸香糖苷。经与文献核对<sup>[11]</sup>, 各种数据基本符合。

化合物 II: 淡黄色针晶, mp 184~185 °C (甲醇), HCl-Mg 反应呈阳性, Molish 反应呈阳性, 分子式: C<sub>28</sub>H<sub>32</sub>O<sub>17</sub>, 进行薄层酸水解, 与标准糖对照, 表明含有葡萄糖。UV λ<sub>max</sub><sup>MeOH</sup> nm: 342, 252, 267 (sh)。ESI-MS m/z: 663.2 (M<sup>+</sup> + Na), 639.2 (M<sup>+</sup> - H)。<sup>1</sup>H-NMR (DMSO-d<sub>6</sub>) δ: 12.55 (1H, s, 5-OH), 10.91 (1H, s, 7-OH), 7.96 (1H, d, J = 2 Hz, H-2'), 7.22 (1H, d, J = 8 Hz, H-5'), 7.53 (1H, dd, J = 2, 8 Hz, H-6'), 6.21 (1H, d, J = 2 Hz, H-6), 6.45 (1H, d, J = 2 Hz, H-8), 5.57 (1H, d, J = 7.5 Hz, H-1''), 5.05 (1H, d, J = 8.5 Hz, H-1''), 3.83 (3H, s, -OCH<sub>3</sub>)。 <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>) δ: 苷元: 157.19 (C-2), 134.13 (C-3), 178.19 (C-4), 161.95 (C-5), 99.53 (C-6), 165.12 (C-7), 94.53 (C-8), 156.43 (C-9), 104.81 (C-10), 124.37 (C-1'), 115.29 (C-2'), 148.83 (C-3'), 149.21 (C-4'), 114.23 (C-5'), 122.15 (C-6'); -Glc: 101.43, 100.27 (C-1), 75.021, 73.84 (C-2), 78.21, 77.81 (C-3), 70.51, 70.28 (C-4), 77.53, 77.12 (C-5), 61.31 (C-6); 56.39 (-OCH<sub>3</sub>)。综合以上数据, 推测该化合物结构为 5, 7-二羟基-3'-甲氧基黄酮醇-3, 4'-O-β-D-二葡萄糖苷。经与文献核对<sup>[12, 13]</sup>, 各种数据基本符合。

化合物 IV: 淡黄色针晶, mp 209~211 °C (甲醇), HCl-Mg 反应呈阳性, Molish 反应呈阳性, 分子式: C<sub>28</sub>H<sub>32</sub>O<sub>17</sub>, 进行薄层酸水解, 与标准糖对照, 表明含有葡萄糖。UV λ<sub>max</sub><sup>MeOH</sup> nm: 357, 252。FAB-MS m/z: 641 [M+H]<sup>+</sup>, 479 [M+H-162]<sup>+</sup>, 317 [M+H-162-162]<sup>+</sup>, 476 (A<sub>1</sub>+162+162)。 <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>) δ: 12.60 (1H, s, 5-OH), 9.82 (1H, s, 4'-OH), 7.95 (1H, d, J = 2 Hz, H-2'), 6.92 (1H, d, J = 8 Hz, H-5'), 7.54 (1H, dd, J = 2, 8 Hz, H-6'), 6.45 (1H, d, J = 2 Hz, H-6), 6.81 (1H, d, J = 2 Hz, H-8), 5.57 (1H, d, J = 6.0 Hz, H-1''), 5.39 (1H, d, J = 8.0 Hz, H-1''), 3.84 (3H, s, -OCH<sub>3</sub>)。 <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>)

δ: 苷元: 157.19 (C-2), 133.97 (C-3), 178.28 (C-4), 160.6 (C-5), 98.50 (C-6), 162.18 (C-7), 94.53 (C-8), 156.71 (C-9), 104.81 (C-10), 121.37 (C-1'), 115.92 (C-2'), 114.21 (C-5'); -Glc: 101.36 (C-1), 75.33 (C-2), 78.18, 77.90 (C-3), 70.52 (C-4), 77.13 (C-5), 61.28 (C-6); 56.39 (-OCH<sub>3</sub>)。综合以上数据, 推测该化合物结构为 4', 5-二羟基-3'-甲氧基黄酮醇-3, 7-O-β-D-二葡萄糖苷。经与文献核对<sup>[14]</sup>, 各种数据基本符合。

化合物 V: 淡黄色针晶, mp 184~185 °C (甲醇), HCl-Mg 反应呈阳性, Molish 反应呈阳性, 分子式: C<sub>28</sub>H<sub>32</sub>O<sub>17</sub>, 进行薄层酸水解, 与标准糖对照, 表明含有葡萄糖。FAB-MS m/z: 641 [M+H]<sup>+</sup>, 479 [M+H-162]<sup>+</sup>, 341.2 [B<sub>2</sub>+162]<sup>+</sup>, 313 [A<sub>1</sub>+162]<sup>+</sup>, 317 [M+H-162-162]<sup>+</sup>, 316 [M-162-162]<sup>+</sup>。 <sup>1</sup>H-NMR (DMSO-d<sub>6</sub>) δ: 12.49 (1H, s, 5-OH), 9.78 (1H, s, 3-OH), 7.78 (1H, d, J = 2 Hz, H-2'), 6.96 (1H, d, J = 8 Hz, H-5'), 7.72 (1H, dd, J = 2, 8 Hz, H-6'), 6.45 (1H, d, J = 2 Hz, H-6), 6.86 (1H, d, J = 2 Hz, H-8), 5.50 (1H, d, J = 3.5 Hz, α-D-H-1''), 5.22 (1H, d, J = 7.0 Hz, β-D-H-1''), 3.85 (3H, s, -OCH<sub>3</sub>)。 <sup>13</sup>C-NMR (DMSO-d<sub>6</sub>) δ: 苷元: 19.73 (C-2), 136.92 (C-3), 176.79 (C-4), 161.10 (C-5), 99.93 (C-6), 163.17 (C-7), 95.42 (C-8), 156.38 (C-9), 104.68 (C-10), 122.62 (C-1'), 116.27 (C-2'), 148.11 (C-3'), 148.07 (C-4'), 112.51 (C-5'), 122.51 (C-6'); -Glc: 105.47, 99.92 (C-1), 76.74, 74.51 (C-2), 77.67, 77.38 (C-3), 70.85, 72.57 (C-4), 77.38, 68.74 (C-5), 61.81, 61.12 (C-6); 56.50 (-OCH<sub>3</sub>)。综合以上数据, 推测该化合物结构为 4', 5, 3-三羟基-3'-甲氧基黄酮醇-7-O-β-D-二葡萄糖苷。经与文献核对<sup>[15]</sup>, 各种数据基本符合。

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## 茶叶中多糖的分离及降血糖活性的研究

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**摘要:**目的 对茶叶中的水溶性中性多糖(NTPS)和酸性多糖(ATPS)进行分离,以探讨不同结构的茶多糖的降血糖活性。方法 采用弱碱性的大孔阴离子交换树脂D315分离出两种带不同电荷的茶多糖NTPS和ATPS,采用气相色谱和离子色谱分析其单糖组成,并采用DEAE-Sepharose FF对其作进一步分离,通过HPGPC法分析其分离产物主要级分的相对分子质量。通过四氧嘧啶ip造成高血糖模型,连续ig茶多糖12d,对比研究两种茶多糖的降血糖活性。结果 NTPS和ATPS都是杂聚糖。NTPS以中性糖为主,含质量分数为6.82%半乳糖醛酸,中性糖部分主要由半乳糖组成;ATPS以酸性糖为主,含质量分数为33.02%半乳糖醛酸,中性糖部分主要由鼠李糖、阿拉伯糖和半乳糖组成。两种茶多糖相对分子质量均小于 $3 \times 10^4$ 。NTPS、ATPS按照200和400 mg/kg·d的剂量连续给四氧嘧啶糖尿病小鼠ig 12 d,发现都能抑制四氧嘧啶糖尿病小鼠血糖的升高。结论 首次采用D315阴离子交换树脂从茶叶中分离出了不同结构的多糖NTPS和ATPS,两种多糖都有很好的降血糖效果。

**关键词:**茶叶;中性多糖;酸性多糖;四氧嘧啶;糖尿病小鼠

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### Isolation of polysaccharides from tea and their hypoglycemic activity

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**Abstract: Objective** To isolate water-soluble neutral polysaccharides (NTPS) and acidic polysaccharides (ATPS) from tea, investigate their hypoglycemic effects, and elucidate the relationship between structure and activity. **Methods** Weak base macroporous anion-exchange resin D315 was used to isolate two kinds of tea polysaccharides NTPS and ATPS based on electric charge of tea polysaccharides. Their monosaccharide compositions were analyzed by gas chromatography and ion chromatography. NTPS and ATPS were further isolated by DEAE-Sepharose FF. Molecular weights of the main fractions were analyzed by HPGPC. The model of diabetic mellitus was established by ip alloxan in mice. The blood samples were collected with tail vein puncture to determine blood glucose. The hypoglycemic activity between neutral and acidic polysaccharides was compared after ig tea polysaccharides for 12 d successively. **Results**

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