Preparation and Determination of 1-O-Acetylbritannilactone in *Inula Britannica* L.

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Abstract

To prepare reference substance for quality control of *Inula Britannica* L., 1-O-acetylbritannilactone was extracted and separated from chloroform extraction of *Inula Britannica* L. Chemical structure of the 1-O-acetylbritannilactone product was elucidated by ultraviolet spectroscopy, UV, IR, infrared spectroscopy, IR, nuclear magnetic resonance, NMR, and mass spectrometry, MS, and the results were in agreement with the reference. The purity of the 1-O-acetylbritannilactone product was 99.5% which satisfies the need of reference substances of traditional Chinese medicines. A method of high performance liquid chromatography-evaporative light scattering detection, HPLC-ELSD, is described for the determination of 1-O-acetylbritannilactone in *Inula Britannica* L. The chromatographic conditions include Hypersil ODS-2 column, a mixture of methanol-water (52:48 V/V) with the flow rate of 1.0 mL/min used as mobile phase. The temperature of drift tube of the ELSD was 90 °C. Flow rate of carrier gas was 2.5 L/min. Linear range of 1-O-acetylbritannilactone was 1.37 to 8.21 μg r = 0.9998. The average recovery of 1-O-acetylbritannilactone was 100.2% with the relative standard deviation, RSD, of 1.3% n = 6. The method is simple, accurate, time saving and reproducible.

Key words high performance liquid chromatography-evaporative light scattering detection

1-O-acetylbritannilactone-reference substance *Inula Britannica* L.

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high performance liquid chromatography-evaporative light scattering detection

1-O-acetylbritannilactone-reference substance *Inula Britannica* L.
酰大花旋覆花内酯的分离和提取

样品购自药材市场。药材经河北医科大学生药教研

1.1 HPLC仪(如K-1500；美国K-1001；UV-K2501)

1.2 1-0-0-0-0 1-0-0-0-0 1-0-0-0-0

1.2.1 1-0-0-0-0 1-0-0-0-0 1-0-0-0-0 2 kg(10)

1.2.2 1-0-0-0-0 1-0-0-0-0 1-0-0-0-0 1 mL

乙酰大花旋覆花内酯的含量，该法准确，简

1.2.3 Zorbax SB-C_{18}，5 mm × 4.6 mm i. d. [5] μm。柱温：30 °C；检测波长：208 nm；

1.2.4 Agilent 1100，DAD

1.2.5 Agilent 1100，DAD

1.3 1-0-0-0-0 1-0-0-0-0

1.3.1 1-0-0-0-0 1.0 g(5)

1.3.2 1-0-0-0-0 105 °C (5)

1.3.3 1-0-0-0-0 1-0-0-0-0 0.5% HCl(7)

2 1-0-0-0-0

2.1 1-0-0-0-0 1-0-0-0-0 1-0-0-0-0

2.2 1-0-0-0-0 1-0-0-0-0 1-0-0-0-0

2.3 1-0-0-0-0 1-0-0-0-0 1-0-0-0-0

2.4 1-0-0-0-0 1-0-0-0-0

2.5 1-0-0-0-0 1-0-0-0-0 1-0-0-0-0 1-0-0-0-0

2.6 1-0-0-0-0 1-0-0-0-0

2.7 1-0-0-0-0 1-0-0-0-0

Fig. 1 Chemical structure of 1-O-acetylbritannilactone
第期 王云志等：氧乙酰大花旋覆花内酯对照品的制备及其在欧亚旋覆花中的含量测定

![图1](image1)

图1 奈特烈科科的纯度测定色谱图

紫外光谱
对照品的甲醇溶液在208 nm处有最大吸收。

![图2](image2)

图2 分子离子峰（见图3）。

质谱
分子离子峰（见图3）。主要碎片离子峰为m/z 308，符合裂解规律（见图3）。

以上<>, >: 89; 9?光谱数据与文献报道值一致，确证对照品为氧乙酰大花旋覆花内酯。

![图3](image3)

图3 分裂图案的质谱图
### 2.3

照品溶液，加甲醇逐步稀释，进样样品溶液。吸取该样品溶液每份次，其乙酰大花旋覆花内酯的保留时间约为取准偏差（以峰面积的对数对质量浓度的对数作图，得线性回

回收率在“方法制备样品溶液。分别加入对照品溶液，“方法制备样品溶液。分别加入对照品溶液

### 2.4

![Chart 1](chart1.png)

**Table 1 The content of 1-O-acetylbritannilactone in I. Britannica L. samples of different sources**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Content mg/g</th>
<th>RSD/%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flowe吉 Shaxi吉</td>
<td>3.44</td>
<td>1.03</td>
</tr>
<tr>
<td>Stef吉 Shaxi吉</td>
<td>2.47</td>
<td>1.26</td>
</tr>
<tr>
<td>Overground pad吉 Shaxi吉</td>
<td>2.62</td>
<td>1.37</td>
</tr>
<tr>
<td>Flowe方 Hebei吉</td>
<td>1.38</td>
<td>1.14</td>
</tr>
<tr>
<td>Flowe方 Henan吉</td>
<td>1.27</td>
<td>1.23</td>
</tr>
</tbody>
</table>

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