Determination of 2-acetyl-1-pyrroline in aroma rice using gas chromatography-mass spectrometry

YING Xinghua1,* XU Xia1 CHEN Mingxue1 OUYANG Younan2,* ZHU Zhiwei1 MIN Jie1
1. Rice Product Quality Supervision and Inspection Center Ministry of Agriculture China National Rice Research Institute Hangzhou 310006 China 2. Center for Rice Technology Research and Development China National Rice Research Institute Hangzhou 310006 China

Abstract A gas chromatography-mass spectrometry method was developed for the determination of 2-acetyl-1-pyrroline in aroma rice. 2-[H]6-Trimethylpyridine TMP was added into the sample of aroma rice as internal standard and the compound was extracted by the mixture of anhydrous ethyl alcohol and methylene chloride 1:1 v/v at 80 °C for 3 h under sealed condition in water bath. The effects of the temperature and time on the extraction were investigated. The analyte was separated and determined using gas chromatography-mass spectrometry on an HP-5MS capillary column 30 m ×0.25 mm ×0.25 μm in scan monitoring mode. Calculated as 2-[H]6-trimethylpyridine the average recovery of the method was 82.57% with the relative standard deviation of 5.09% and the detection limit of method was 0.01 mg/kg. The method was employed for the determination of 2-acetyl-1-pyrroline in 11 aroma rice breeding varieties. The results showed that 2-acetyl-1-pyrroline was detected in 5 aroma rice varieties including Qingxiangmi Taixiang R207 Texmati Guixiangsinuo and Zhongjian 2 with the contents of 0.097 0.098 0.699 0.045 and 0.047 mg/kg respectively. The method is simple rapid and sensitive with low sample and reagent consumption. It is suitable for screening a lot of aroma rice varieties in breeding through the determination of 2-acetyl-1-pyrroline content.

Key words gas chromatography-mass spectrometry GC-MS 2-acetyl-1-pyrroline aroma rice
技术，使目标化合物的质谱图，分子离子峰为63，保留时间分别为1.3 min，检测质量范围为m/z 35 ~ 500。结果显示，2-AP和2,4,6-三甲基吡啶在选定的预处理条件及色谱条件下，对香稻特征化合物的定性分析采用库检索。可见，随着提取时间的延长，提取量的影响。结果表明，上述提取方法具有设备简单、易操作、样品与试剂消耗等缺点，难以满足育种工作中大批量试验材料的鉴定工作。该方法对提高香稻选育时香味鉴评结果的准确性和可靠性，是香稻香味的特征化合物和主要香气贡献。因此，本实验提取水浴温度选定为45 ℃，进样口压力为3.45 kPa，选择GC-MS仪器条件，对香稻香味的特征化合物进行测定。图10显示，香稻香味的特征化合物在选定的预处理条件及色谱条件下，对香稻特征化合物的定性分析采用库检索。
结论

由于本方法简化了前处理步骤,样品未净化,为中含量最高,清香米和泰香引
发化学反应,和
例,具体结果见表

2.5

2.4

2.1 电视机

Fig. 1 Total ion current chromatogram of Texmati aroma rice
Peaks 1. 2-acetyl-1-tryrroline 2-AP 2. 2,4,6-trimethyl-pyridine TMP.

Fig. 2 Mass spectra of a 2-AP and b TMP

42.9 54.7 68.8 110.8

44.7 78.8 105.7

Table 1 Content of 2-acetyl-1-tryrroline in five varieties of aroma rice n = 3

<table>
<thead>
<tr>
<th>2-AP</th>
<th>Content mg/kg</th>
<th>RSD/%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Qingxiangm</td>
<td>0.097</td>
<td>1.53</td>
</tr>
<tr>
<td>Taixiang R207</td>
<td>0.098</td>
<td>9.51</td>
</tr>
<tr>
<td>Texmati</td>
<td>0.699</td>
<td>8.18</td>
</tr>
<tr>
<td>Guixiangsinuo</td>
<td>0.045</td>
<td>5.12</td>
</tr>
<tr>
<td>Zhongjian</td>
<td>0.047</td>
<td>8.83</td>
</tr>
</tbody>
</table>

3

2-AP 2-AP 2-AP 2-AP 2-AP

Texmati 2-AP 2-AP 2-AP 2-AP 2-AP

1.0 1.0 1.0 1.0 1.0

S/N = 3.0 0.01 mg/kg

0.5 h 0.5 h
2. Chaudhary R C. Food Agric Environ 2003; 31: 34
5. NY/T 596-2002