Determination of three adrenergic drugs using capillary electrophoresis with amperometric detection

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Abstract A method for the determination of three adrenergic drugs including phenylephrine hydrochloride PHE metaraminol bitartrate MR and isoprenaline hydrochloride IP was developed using capillary electrophoresis with amperometric detection. The detection potential of working electrode was 0.950 V versus the reference electrode of Ag/AgCl. At the applied voltage of 18 kV the three analytes were completely separated within 18 min in 50 mmol/L borate buffer pH 10.00 with the injection time of 10 s. Good linear relationships were obtained for all the three analytes in the range of 2 – 100 μmol/L. The detection limits for PHE MR and IP were 0.8 – 0.8 and 1.0 μmol/L respectively. The proposed method was applied to the analysis of some injection drugs and the results were satisfactory.

Key words capillary electrophoresis CE amperometric detection adrenergic drugs
phenylephrine hydrochloride, metaraminol bitartrate, isoprenalin hydrochloride, PHE, MR, IP.

1.3 CE Conditions

10 min, 18 kV, 5 min, 18 kV, 0.1 mol/L NaOH, 15 min, 18 kV, 0.1 mol/L NaOH, 20 min, KCl, pH 10.00, + 0.950 V vs. Ag/AgCl.

1.4 Samples

PHE, MR, IP.

2.1 Buffers

CV, PHE, MR, IP, 0.1 mol/L NaOH, 0.1 mmol/L PHE, MR, IP, CV, pH 10.00, 0.1 V, 3 min, PHE, MR, 0.6 V, 0.950 V.

2.2 Introduction
图 2 拟肾上腺素药物的循环伏安曲线

图 3 散射的 pH 对 MR、PHE 和 IP 的分离影响

缓冲溶液浓度的影响

缓冲溶液的浓度会影响分析物的迁移时间，从而影响分离度。本实验考察了硼酸盐缓冲液浓度在范围内（括号内的数值）对 MR、PHE 和 IP 的分离影响，结果见图 4。由图 4 可知，当缓冲溶液的浓度小于 20 mmol/L 时，MR、PHE 和 IP 的分离效果较差。随着缓冲液浓度的增加，MR、PHE 和 IP 可以得到较好的分离。但考虑缓冲液浓度太大时，会出现宽展尾现象，故本实验选择硼酸盐缓冲溶液的浓度为 50 mmol/L。
2.2.3 \[\text{PHE} \quad \text{MR} \quad \text{IP}\]

Fig. 5 Effect of separation voltage on the separation of PHE, MR, and IP

Conditions: concentrations of PHE, MR, IP, 0.1 mmol/L; working potential + 0.950 V vs. Ag/AgCl; injection time 10 s; running buffer 50 mmol/L Na₂B₄O₇, pH 10.00.

2.2.4 \[\text{PHE} \quad \text{MR} \quad \text{IP}\]

18 kV \[5 \sim 15 \text{ s}\]

2.2.5 \[\text{PHE} \quad \text{MR} \quad \text{IP}\]

0.1 mmol/L \[n = 3\]

RSD \[0.72\% \sim 1.50\% \quad 5.63\% \sim 7.80\%\]

Table 1 Reproducibilities of migration times and peak areas of PHE, MR, IP \[n = 3\]

<table>
<thead>
<tr>
<th>Compound</th>
<th>Migration time</th>
<th>Peak area</th>
</tr>
</thead>
<tbody>
<tr>
<td>PHE</td>
<td>0.72</td>
<td>5.63</td>
</tr>
<tr>
<td>MR</td>
<td>0.85</td>
<td>7.80</td>
</tr>
<tr>
<td>IP</td>
<td>1.50</td>
<td>6.85</td>
</tr>
</tbody>
</table>

2.3 \[\text{PHE} \quad \text{MR} \quad \text{IP} \quad n = 3\]

Table 2 Regression equations and detection limits of PHE, MR, and IP

<table>
<thead>
<tr>
<th>Compound</th>
<th>Regression equation</th>
<th>Linear range/µg/L</th>
<th>Detection limit/µg/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>PHE</td>
<td>(Y = 6 \times 10^{10}X - 5547)</td>
<td>0.9999</td>
<td>0.41 \sim 20.4</td>
</tr>
<tr>
<td>MR</td>
<td>(Y = 4 \times 10^{10}X - 953)</td>
<td>0.9999</td>
<td>0.63 \sim 31.7</td>
</tr>
<tr>
<td>IP</td>
<td>(Y = 4 \times 10^{10}X - 10413)</td>
<td>0.9991</td>
<td>0.50 \sim 24.8</td>
</tr>
</tbody>
</table>

\(Y\) peak area \(X\) mass concentration \(\mu\)g/L.

Fig. 6 Electropherogram of a mixed solution of PHE, MR, and IP standards

Conditions: concentrations of PHE, MR, IP, 0.1 mmol/L; working potential + 0.950 V vs. Ag/AgCl; separation voltage 18 kV; running buffer 50 mmol/L Na₂B₄O₇, pH 10.00; injection time 10 s.

1. PHE; 2. MR; 3. IP.

2.3 \[\text{PHE} \quad \text{MR} \quad \text{IP} \quad n = 3\]

Table 3 Determination of the samples \(n = 3\)

<table>
<thead>
<tr>
<th>Injection</th>
<th>Original/µg/L</th>
<th>Added/µg/L</th>
<th>Found/µg/L</th>
<th>Recovery/%</th>
<th>RSD/%</th>
</tr>
</thead>
<tbody>
<tr>
<td>PHE</td>
<td>10.00</td>
<td>10.00</td>
<td>9.99</td>
<td>99.9%</td>
<td>5.2</td>
</tr>
<tr>
<td></td>
<td>10.00</td>
<td>9.64</td>
<td>96.4%</td>
<td>4.5</td>
<td></td>
</tr>
<tr>
<td>MR</td>
<td>10.00</td>
<td>10.69</td>
<td>106.9%</td>
<td>3.5</td>
<td></td>
</tr>
<tr>
<td></td>
<td>10.00</td>
<td>10.96</td>
<td>109.6%</td>
<td>2.9</td>
<td></td>
</tr>
</tbody>
</table>

\(n = 3\)
Fig. 7 Electropherogram of a PHE injection