Colorimetric Method for the Determination of Piperazine in Pharmaceutical Formulations

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Piperazine and its salts are reacted with alcoholic p-benzoquinone and buffered at pH 5.4 to give a coloured product with maximum absorption at 516 nm. The piperazine base has a molar absorptivity of 0.96 × 10^4 1/mol/cm, and Beer’s law is obeyed in the range of 4-20 µg/ml. When applied to two commercial preparations, the proposed method gave mean recoveries within 1%, and relative standard deviation was less than 1%.

This paper presents a simpler direct method for the determination of piperazine by reaction with p-benzoquinone at room temperature without prior separation of the free base. The method is very suitable for routine analysis and can replace the official gravimetric method, which is based on precipitating as dipicrate salt. A Shimadzu 1601 UV/Vis spectrometer with 1 cm matched quartz cells was used for all absorbance measurements. All other chemicals used were analar grade. P-benzoquinone solution (1% w/v) in 95% ethanol and buffer solution (pH 5.4) were freshly prepared. Stock solution of piperazine citrate was prepared by dissolving 100 mg in 100 ml of distilled water. The stock solution was further diluted to get a working standard solution containing 80 µg/ml. In a series of 20 ml volumetric flasks, 1.0 ml to 5.0 ml of standard piperazine citrate solution was transferred separately; and to each flask, 2 ml of buffer solution (pH 5.4) and 2 ml of p-benzoquinone solution were added. This was allowed to stand for 30 min and diluted to volume with distilled water. The absorbance of the coloured solutions was measured at 516 nm against reagent blank.

Two grams of standard piperazine citrate was added to previously analyzed tablets and syrup and solution so obtained was treated as described above. Absorbance of the coloured solution (80 µg/ml) was measured at 516 nm at intervals of 5 min till the end of 30 min. The effect of pH of the buffer used has been examined, and optimum pH for high sensitivity, minimal blank reading and high stability was found to be 5.4. The colour

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TABLE 1: ESTIMATION OF PIPERAZINE IN PHARMACEUTICAL FORMULATIONS

<table>
<thead>
<tr>
<th>Pharmaceutical formulations</th>
<th>Labeled amount of piperazine citrate (mg)</th>
<th>Amount of piperazine citrate found (mg)*</th>
<th>Amount added (mg)*</th>
<th>Recovery studies</th>
</tr>
</thead>
<tbody>
<tr>
<td>Piperazine citrate syrup</td>
<td>750 / 5 ml</td>
<td>738</td>
<td>2</td>
<td>735</td>
</tr>
<tr>
<td>Antipar tablets</td>
<td>500</td>
<td>490</td>
<td>2</td>
<td>501</td>
</tr>
</tbody>
</table>

*Average of six readings

TABLE 2: COMPARISON OF RECOMMENDED PROCEDURE WITH THE OFFICIAL BP METHOD

<table>
<thead>
<tr>
<th>Sample</th>
<th>Recovery ± S.D., %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Official method</td>
</tr>
<tr>
<td>Piperazine citrate syrup</td>
<td>97.2±0.5</td>
</tr>
<tr>
<td>Antipar tablets</td>
<td>99.2±0.4</td>
</tr>
</tbody>
</table>

*Average of six readings

previously methods, viz., no heating is required. Since p-benzoquinone reacts with primary, secondary and cyclic and heterocyclic amines, any of these compounds, if present, might interfere with the determination. However, application of the molar ratio and continuous variation methods under the conditions given in this paper showed the reaction ratio of p-Benzoquinone to piperazine to be 4:1.

REFERENCES