Assay Determination of Rabeprazole Pellets Dosage forms by HPLC

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Abstract: Rabeprazole is a Bezimidazole derivative. It works as proton pump inhibitor, an anti-ulcerative drug used against peptic ulcer syndrome to suppress excess acid discharge in the stomach. A simple, accurate, sensitive and precise High performance liquid chromatography method was proposed for the determination of Rabeprazole. The solutions of sample and standard were prepared in 0.1 N NaOH. In the High performance liquid chromatography method, the quantitative determination of the drug was carried at 280 nm and the linearity range was found to be 14-26 μg/mL. The calibration graphs were constructed at their wavelength, and were found to be linear for HPLC methods. The proposed methods have been validated that included parameters such as linearity, accuracy, precision, LOD, LOQ, recovery and robustness, no significant difference between the performance of the proposed method concerning the mean values and normal deviations. The proposed methods can be utilized for analysis of pharmaceutical formulation.

Keywords: Proton pump inhibitor, Peptic ulcer, High performance liquid chromatography, calibration graphs, validation, Rabeprazole

Rabeprazole Sodium Synthesis Flow Chart

1. Introduction

Rabeprazole is a Bezimidazole derivative. It works as proton pump inhibitor, an anti-ulcerative drug used against peptic ulcer syndrome to suppress excess acid discharge in the stomach. A simple, accurate, sensitive and precise high performance liquid chromatography method was proposed for the determination of Rabeprazole. The solutions of sample and standard were prepared in 0.1 N NaOH. Structure of Rabeprazole as shown below

Rabeprazole structure

2. Chemicals and Instruments

Potassium di hydrogen orthophosphate, Sodium hydroxide, Rabeprazole sodium working standard, shimadzu LC solutions 2010 software Double distilled water.

3. Method

Chromatographic conditions
Column: 4.6mm3D×25cm long. Packed with C18 with particle size 5μ
Flow rate: 1.0ml/min
Wave length: 280nm
Injection volume: 20μl

Buffer pH 7.4: Dissolve 6.8 grams of Potassium di hydrogen orthophosphate and 1.5 grams of sodium hydroxide in 1000 ml water adjust pH 7.4 with 0.1 M NaOH. Mobile phase: Prepare a suitable quantity of a filtered and degassed mixture of 65 volume of Phosphate buffer pH 7.4, 35 volume of acetonitrile

Standard Preparation: Transferred an accurately weighed quantity of about 20mg of Rabeprazole Sodium working standard to a 100 ml volumetric flask. Add 40ml of 0.1M sodium hydroxide and sonicate to dissolve. Make volume up to the mark with 0.1M sodium hydroxide and mix. Transfer 5 ml of the solution in to 50ml volumetric flask make up with mobile phase and mix.

Sample preparation: Take around 5 grams of pellets in to a mortar and pestle and grind the pellets in to a uniform fine powder. Weigh accurately a bout a quantity equivalent to 20 mg of Rabeprazole sodium in to a dry 100 ml volumetric
flask add 50 ml of 0.1 M sodium hydroxide and sonicate to dissolve. Make volume up to the mark with 0.1M sodium hydroxide and filter 20to 30ml of solution into dry test tube. Further dilute 5ml of the filtrate to 50ml with mobile phase.

**Procedure:** Inject sample preparation in duplicate in to the chromatograph and record the chromatograms. Measure the response for the major peaks. Calculate the quantity in percentage w/w of Rabeprazole sodium by using below formula.

\[
\frac{AS \times WS \times \text{sampile dilution}}{SA \times \text{standard dilution} \times \text{sample weight}} \times PS = \text{Assay}\%
\]

\[
\frac{AS \times WS \times 5 \times 100}{SA \times 50 \times 5} \times PS = \text{Assay}\%
\]

\[
\text{PS} = \text{Purity of working standard}
\]

\[
\text{SA} = \text{Average peak area of sample preparation}
\]

\[
\text{Aw} = \text{Average weight of sample taken in mg}
\]

The results are tabulated as follows

<table>
<thead>
<tr>
<th>Semi formulation</th>
<th>S. No</th>
<th>Label claim</th>
<th>Amount estimated</th>
<th>%label claim</th>
<th>% Deviation</th>
<th>S.D</th>
<th>RSD</th>
</tr>
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<tbody>
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<td>Pellets</td>
<td>1</td>
<td>8.5%</td>
<td>8.52</td>
<td>100.2353</td>
<td>0.2353</td>
<td>0.20265</td>
<td>0.5439</td>
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<td>100.7059</td>
<td>0.7059</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
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<td>100.3529</td>
<td>0.3529</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
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<td>8.51</td>
<td>100.1176</td>
<td>0.1176</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td></td>
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<td>100.4706</td>
<td>0.4706</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>6</td>
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<td>8.53</td>
<td>100.3529</td>
<td>0.3529</td>
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<td></td>
</tr>
</tbody>
</table>

**4. Method Validation**

Above method is validated by considering the validation parameters such as linearity, accuracy, precision, LOD, LOQ, recovery and robustness, no significant difference between the performance of the proposed method concerning the mean values and normal deviations. The linearity range was found to be 14-26 μg/ml.

**Linearity**

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<tr>
<td>2</td>
<td>0.017</td>
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<tr>
<td>3</td>
<td>0.02</td>
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<tr>
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<td>0.023</td>
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<tr>
<td>5</td>
<td>0.026</td>
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**5. Result and Discussion**

System suitability test is applied to check the various parameters such as efficiency, resolution and asymmetry. The results obtained are shown in the table that is in concurrence with the USP requirement

<table>
<thead>
<tr>
<th>S. No</th>
<th>Parameter</th>
<th>Rabeprazole</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Theoretical plate</td>
<td>8250</td>
</tr>
<tr>
<td>2</td>
<td>Tailing factor</td>
<td>1.928</td>
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<tr>
<td>3</td>
<td>RSD for 6 injections</td>
<td>0.5439</td>
</tr>
</tbody>
</table>

**References**


[3] ICH Guidelines
