Qualitative and Quantitative Analysis of Paracetamol in Different Drug Samples by HPLC Technique

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Abstract: HPLC is rapid more accurate, purity checking & separation method of paracetamolin different drug sample as compare with standard reference & sample solution within complies with Indian Pharmacopeia. This method is applicable to Forensic science laboratories also. It is very sensitive less time consumable but sharp peaks recorded. Chromatographic technique – stainless still column 20cm x 4.6mm packed with ocadecysilance bonded to porous Silica 10 micrometer. Flow rates 0.2ml/min. at 20 micrometer loop injector & spectrophotometer set at 272 nm. Retention time of std 4-amino phenol & sample solution 4.388 & 4.542 respectively. Labeled amount 102.86% of paracetamol was observed. The % is indicated that the paracetamol is within limit of Indian pharmacopeia.

Identification: Sample given Indian pharmacopeia identification test for the presence of paracetamol.

KeyWords: Analysis, Chromatogram, Drug Sample, HPLC, Paracetamol, Retention time, Spectrophotometer

I. Introduction

Paracetamol, N(4hydroxy phenyl acetamide) is analgesic and antipyretic drug. It is used to prevent fever, headaches & colds. It is one of the major drug & also used along with other drug. Paracetamol is part of the class of drug known as “aniline analgesic”. So it is need to analyze paracetamol quantitatively & qualitatively. A lower dosage of additional non steroid anti-inflammation drug to be used by minimizing over all side effect [1-3] Present work includes analysis of paracetamol. It is active metabolite of phenacetin & not considered to be carcinogenic at therapeutic doses. Main aim of this study was to develop a new RP-HPLC [4-6] method for determination of sample and standard paracetamol from formulated tablet. This method has been reported for the analysis of paracetamol & its combination in pharmaceuticals or in biological fluids. Paracetamol has been determined in combination with other drug’s using UV–spectrophotometry [7-10]. Paracetamol (PARA) is chemically N-(4-hydroxy phenyl) acetamide (amide derivative). It functioned as a weak inhibitor of the synthesis of prostaglandins (PGs) [11]. However, in vivo effects of paracetamol are similar to those of the selective cyclooxygenase-2 (COX-2) inhibitors [12]. Paracetamol also decreases PG concentrations in vivo. Structure of Paracetamol (PARA) is shown in Fig. 1.

II. Experimental

Identification: Sample given Indian pharmacopoeia identification test for the presence of paracetamol.

Paracetamol test for 4-aminophenol

Chromatographic technique
Stainless steel column 20cm x 4.6mm packed with ocadecysilance bonded to porous Silica 10 micrometer.

Mobile phase:
0.01M Sodium Butanesulphate in a mixture of 85 volume of water,
15 volume of methanol and 0.4 volume of formic acid
Flow rate 0.2 ml per minute.
Spectrometer set at 272nm.
At 20 micrometer loop injector

**Preparation of standard test solution:**
Approximately 4-5 gm of standard 4-aminophenol was taken in silica crucible covered with glass funnel and evaporated with continually. A sublimate of white powder was formed. Accurately 0.25mg of sample was taken in 1000ml standard flask and 15% methanol was added up to the mark. 1 ml of this solution was taken in 25ml standard flask diluted with methanol up to the mark. Filter this solution. Sonicator & then used for HPLC. Peak was recorded as shown in Fig. 2.

**HPLC curve of Standard 4- amino phenol figure 2**

**Preparation of paracetamol Sample solution (C₆H₇NO):**
1.187gm of paracetamol was taken in 100ml standard flask & 15% methanol were added up to the mark and than solution was sonicated. This solution was filtered and then used for HPLC. The chromatogram was recorded as shown in fig. 3.

**HPLC curve Sample solution of 4- amino phenol figure 3**
III. Result & discussion

In chromatogram obtained with the test solution, the area of any peak corresponding to 4-aminophenol is not greater than the area of peak in the chromatogram obtained with the reference solution. In the chromatogram obtained with the test solution peak with long retention time.

Uniformity weight of tablets
Sample complies with Indian pharmacopeia requirement for uniformity weight of paracetamol tablet 1.

<table>
<thead>
<tr>
<th>Sr. no.</th>
<th>weight of paracetamol tablet (gm)</th>
<th>In gram</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.5974</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.5996</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>0.5947</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>0.5858</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>0.5993</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>0.5900</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>0.5876</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>0.5857</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>0.5962</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>0.6065</td>
<td></td>
</tr>
</tbody>
</table>

Different weight of paracetamol as shown in table 2

<table>
<thead>
<tr>
<th>Sr. no.</th>
<th>weight of 20 tablets (gm)</th>
<th>In gram</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>11.861</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.593</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>0.029</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>0.563</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>0.622</td>
<td></td>
</tr>
</tbody>
</table>

Calculation:

Weight of paracetamol = sample area / standard area x standard weight / standard dilution / sample weight x average weight x purity of sample.

\[
= \frac{0.5468}{0.715} \times 10/1000 \times 20/0.1762 \times 0.5931 \times 100
\]

\[
= 0.764 \times 0.01 \times 1135.07 \times 10 \times 10 \times 0.5931
\]

\[= 514.33 \text{mg of paracetamol} \]

\[= 514.33/500 \times 100 \]

Weight of paracetamol = 102.86% of label amount.
I.P. limit 95% to 105% of label amount)

IV. Dissolution of Paracetamol tablets:

Sample complies with Indian pharmacopeia requirement for dissolution test.

27.8gm of standard paracetamol was taken in 50ml standard flask and diluted with water. 1ml of this solution was taken in another 50ml standard flask and diluted up to the mark, using distilled water.

6 tablets was taken for sampling each tablet was dissolved in separate 900 ml of distilled water and phosphate buffer PH 5.8 further it was dissolved for 30 minute at 50 RPM, it becomes totally dissolved. 1ml of this solution was taken in 50 ml standard flask and dilute with distilled water up to the mark. Absorbance was measured at 243 nm as shown in fig. 4.
V. Conclusion:

On the basis of above observation, measured sample solution at 243 nm. UV- Spectrophotometer the concentration of ParacetamolRs. Calculated the content of paracetamol was not less than 80% of the standard amount of Paracetamol. The observed percentage is indicated that the Paracetamol is within the limit of Indian pharmacopeia. The Paracetamol sample is indicated that there is rare side effect in the human body may be formed. High speed analysis, less time consumable checking purity of sample & more intense peak recorded in RP-HPLC technique. It can be simultaneous determination for standard reference solution & sample solution of Paracetamol.

Acknowledgment

I would like to thanks Dr.Rajendra P. Pawar, Associate Professor & Head, Department of Chemistry, Deogiri College Aurangabad, Dr. SuryakantSapkal Asst. Prof. JNEC College, Aurangabad & Dr.Prashant Ghosh, Dr. SujataPatil, Mr. Maruti Saundade with Cooperative Institute of MSS’s College of Engineering & Technology, Nagewadi.JALNA.

References