Visible Spectrophotometric Estimation of Aceclofenac and Indapamide from Tablets using Folin-Ciocalteu Reagent

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Two simple, accurate, fast and economical methods have been developed for the quantitative estimation of aceclofenac and indapamide from their respective tablet formulation using Folin-Ciocalteu reagent. Aceclofenac forms a blue colored chromogen with the reagent, which shows absorbance maxima at 642.6 nm and linearity in the concentration range of 80-160 µg/ml of drug. Indapamide forms a green colored chromogen with the reagent, which shows absorbance maxima at 783.2 nm and linearity in the concentration range of 2-12 µg/ml of drug. The results of analysis for both the methods were validated statistically and by recovery studies.

Aceclofenac, chemically [(2-[2-(2,6-Dichlorophenyl)aminophenyl]acetyl)oxy]acetic acid, is a new analgesic and antiinflammatory drug used in the management of osteoarthritis, rheumatoid arthritis and ankylosing spondylitis. It is official in BP, which describes a liquid chromatographic method for its quantitation. Literature survey reveals spectrophotometric, HPLC, spectrofluorometric and densitometric methods for the estimation of aceclofenac from pharmaceutical formulation. Indapamide, chemically 4-chloro-N-[(2RS)-2-methyl-2,3-dihydro-1H-indole-1-yl]-3-sulphamoyl benzamide is a diuretic drug, widely employed in the treatment for hypertension and edema associated with heart failure. It is official in BP and USP, both pharmacopoeia describe a TLC method for its quantitation. Literature survey reveals two derivative spectrophotometric, HPLC, spectrofluorometric and densitometric methods for the estimation of indapamide from pharmaceutical formulations.

In the present work an attempt has been made to develop new colorimetric methods for the estimation of aceclofenac and indapamide from their respective tablet formulation. The developed methods are based on the formation of blue and green colored chromogen with Folin-Ciocalteu (FC) reagent. Commercial FC reagent (CDH) was diluted 1:2 with double distilled water and sodium hydroxide solution (0.1 N and 0.5 N) were prepared in double distilled water. A Systronic double beam UV/Vis spectrophotometer model 2101 with 1 cm matched quartz cells was used for absorbance measurement.

Standard solution of aceclofenac was prepared by dissolving 100 mg in 100 ml of methanol to give stock solution of concentration 1 mg/ml of drug. To a series of 25 ml volumetric flasks, volumes of standard aceclofenac solution (1 mg/ml) ranging from 2.0 to 4.0 ml was transferred, then 10 ml of 0.1 N NaOH and 1 ml diluted FC (1 N) were added to each flask and mixed well. After five minutes, volume was made up to 25 ml with 0.1 N NaOH. The absorbance of blue colored species was measured at 642.6 nm against a reagent blank within 20 min. A calibration curve was constructed.

Standard solution of indapamide was prepared by dissolving 10 mg in 100 ml of 0.5 N NaOH to give stock solution of concentration 100 µg/ml of drug. To a series of 10 ml volumetric flasks, volumes of standard indapamide solution (100 µg/ml) ranging from 0.2 to 1.2 ml were added. Volume was made up to 10 ml with 0.5 N NaOH. To 10 ml of final dilution taken in a test tube added 5 ml of diluted FC reagent and kept for 5 min. The absorbance of green colored species was measured at 783.2 nm against a reagent blank with in 20 min. Another calibration curve was constructed.

For analysis of aceclofenac, twenty tablets were accurately weighed and powdered. The powder equivalent to 100 mg of aceclofenac was weighed accurately and mixed with 50 ml of methanol. The mixture was allowed to stand for 15 min with intermittent shaking.

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The mixture was then filtered through a Whatman filter paper No. 40 and the residue was washed thoroughly with methanol. The filtrate and washings were combined in 100 ml volumetric flask and volume was made up to the mark with methanol. The sample solution was treated as per the procedure described for the standard solution and the amount of aceclofenac present in the solution was computed from calibration curve. Results of analysis are reported in Table 1.

For indapamide, twenty tablets were accurately weighed and powdered. The powder equivalent to 10 mg of indapamide was weighed accurately and mixed with 0.5 N NaOH. The mixture was allowed to stand for 15 min with intermittent shaking. The mixture was then filtered through a Whatman filter paper No. 40 and the residue was washed thoroughly with 0.5 N NaOH. The filtrate and washings were combined in a 100 ml volumetric flask and volume was made up to the mark with 0.5 N NaOH. The sample solution was treated as per the procedure described for the standard solution and the amount of aceclofenac present in the solution was computed from calibration curve. Results of analysis are reported in Table 1.

In order to justify the reliability and suitability of the proposed method, recovery studies were performed by adding known amounts of respective drug to its various preanalyzed formulations at three different concentration levels. The mixtures were analyzed by the proposed methods. The results of recovery studies are reported in Table 1.

The concentration of NaOH and FC reagent, sequence of addition of reagents, the reaction time and final dilutions were selected as a compromise between optimum sensitivity, stability and minimum blank reading. The sample solution of aceclofenac shows maximum absorbance at 642.6 nm and sample solution of indapamide shows maximum absorbance at 783.2 nm. Beer’s law was obeyed in the concentration range of 80-160 µg/ml (r = 0.9978) for aceclofenac and 2-12 µg/ml (r= 0.9993) for indapamide. The percentage recovery values were close to 100%, which indicates that there was no interference of excipients. The proposed methods were precise, sensitive and hence could be used for routine analysis of drugs from tablet formulations.

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REFERENCES


### TABLE 1: RESULT OF ANALYSIS AND RECOVERY STUDIES

<table>
<thead>
<tr>
<th>Drug</th>
<th>Label claim mg/tab</th>
<th>% Label claim estimated*</th>
<th>% Recovery**</th>
<th>Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aceclofenac</td>
<td>100</td>
<td>98.70</td>
<td>98.70</td>
<td>0.195</td>
</tr>
<tr>
<td>Indapamide</td>
<td>2.5</td>
<td>98.50</td>
<td>98.95</td>
<td>0.529</td>
</tr>
</tbody>
</table>

*Average of five determinations. **Average of recovery studies at three different concentration levels