UV Spectroscopic and Colorimetric Methods for the Estimation of Gatifloxacin in Tablet Dosage Forms

K. ILANGO*, P. VALENTINA, K. S. LAKSHMI, ARVIND CANHEA, SAPANA RACHEL ABRAHAM, V. BHASKAR RAJU AND A. KIRAN KUMAR
Department of Pharmaceutical Chemistry, S.R.M. College of Pharmacy, Kattankulathur-603 203, India.

Two simple and sensitive spectroscopic methods in ultra violet and visible region, were developed for the estimation of gatifloxacin in pharmaceutical dosage forms. Method A is based on gatifloxacin, showing absorption maximum at 295 nm, in methanol. The method B is based on the reaction of gatifloxacin, with 0.2% w/v 3-methyl-2-benzothiazolinone hydrazone reagent in presence of 1% w/v ferric chloride solution, to yield a yellow orange colour. This colour has a characteristic light absorption in the visible region, with absorption maximum at 433 nm. These methods obey Beer's law in the concentration range of 2 to 10 µg/ml and 50 to 150 µg/ml, respectively. The proposed method is precise, accurate, and reproducible, and can be extended to the analysis of gatifloxacin in tablet formulations.

*For correspondence
E-mail: kilango67@yahoo.com
Chemically, gatifloxacin is \((\pm)\) 1-cyclopropyl-6-fluoro-1,4-
dihydro-8-methoxy-7-(3-methyl-1-piperazinyl)-4-oxo-3-
quinoinecarboxylic acid sesquihydrate. It is a synthetic
broad-spectrum 8-methoxyfluroquinolone antibacterial
drug, used in the treatment of community-acquired pneumonia,
acute bacterial sinuitis, acute bacterial exacerbation of chronic bronchitis,
and complicated and uncomplicated urinary tract infections. It acts
intravenously by inhibiting topoisomerase II (DNA gyrase) or
topoisomerase IV. It is not official in any pharmacopoeia. A survey of literature, reveals that
 gatifloxacin has been estimated in plasma by HPLC, LC-
MS and HPTLC methods. No spectrophotometric
methods are cited in the literature. We report two simple
and sensitive spectrophotometric methods for the
analysis of gatifloxacin from pharmaceutical dosage forms.

A Systronic UV/Visible spectrophotometer model 119
with 1 cm matched quartz cell, was used for all the
absorbance measurements. All the reagents used were
of analytical grade. Aqueous solution of MBTH (0.2%
w/v) and ferric chloride (1% w/v) were prepared
freshly. A standard solution of gatifloxacin containing
1 mg/ml, was prepared by dissolving pure 100 mg
gatifloxacin in 100 ml of methanol for method A, and
distilled water for method B. It was further diluted to a
concentration of 1 µg/ml for method A, and 100 µg/ml
for method B.

In method A, aliquots of working standard gatifloxacin (1
to 5 ml) solutions were transferred into a series of 10 ml
volumetric flask, and the volume was made up to 10 ml
with methanol. The absorbance of each solution was
measured at 295 nm against methanol as blank.

In method B, aliquots (0.5 to 3 ml) of the solutions were
taken into 10 ml volumetric flasks, and 1 ml of ferric
chloride solution was added, followed by 1 ml of MBTH
reagent, and kept aside at room temperature for 10 min.
The absorbance of the resulting yellow orange colour
chromogen was measured at 433 nm against the reagent
blank.

Twenty tablets of gatifloxacin (Gatiquin 200 mg tablets from
Cipla, Ahmedabad and Gaity 200 mg tablets from Dr.
Reddy’s Labs, Hyderabad), were weighed accurately and
powdered. The amount of powder equivalent to 100 mg
of gatifloxacin, was weighed and dissolved in respective
solvents to make 100 ml, and filtered through Whatmann
filter paper No. 41. The filtrate was further diluted to 1
µg/ml for method A, and 100 µg/ml for method B. The
amount of gatifloxacin present in tablets were estimated
by interpolation, from the calibration curve.

The optical characteristics such as Beer’s law limits,
Sandell’s sensitivity, molar absorptivity, and correlation
coefficient for the proposed two methods, are summarised
in Table 1. The recovery studies were carried out to
ascertain the accuracy and precision of the proposed
method, by adding a known amount of standard solution at
three levels to the previously analyzed sample solution,
and the absorbance was measured. The results obtained
by the proposed methods were in good agreement with
the labeled amount (Table 2).

In method A, gatifloxacin exhibited λmax at 295 nm
in methanol. Method B is based on the structure of
gatifloxacin, the piperazine nitrogen, that may be
subjected to oxidation to form stable nitroxides, which
gives a yellow orange complex with MBTH reagent.
Stability of the colour complex was determined by
measuring absorbance of the chromogen at specified time
intervals, and was found to be stable for 2 hr. These
results indicate, that the proposed methods are simple,
sensitive, accurate, and reproducible, and can be
employed for routine quality control analysis of

### Table 1: Optical Characteristics and Precision

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Method A</th>
<th>Method B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Absorption maxima</td>
<td>295 nm</td>
<td>433 nm</td>
</tr>
<tr>
<td>Beer’s law limit (µg/ml)</td>
<td>2 to 10</td>
<td>50 to 150</td>
</tr>
<tr>
<td>Molar absorptivity (l/mol/cm)</td>
<td>3.069 × 10³</td>
<td>2.293 × 10³</td>
</tr>
<tr>
<td>Sandell’s Sensitivity (µg/cm/0.001)</td>
<td>0.0122</td>
<td>0.164</td>
</tr>
<tr>
<td>Regression equation (Y = mx+c)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Slope (m)</td>
<td>8.01 × 10⁻³</td>
<td>5.6 × 10⁻³</td>
</tr>
<tr>
<td>Intercept (C)</td>
<td>6.9 × 10⁻³</td>
<td>4.4 × 10⁻³</td>
</tr>
<tr>
<td>Correlation coefficient (r)</td>
<td>0.9997</td>
<td>0.9994</td>
</tr>
</tbody>
</table>

### Table 2: Analysis of Gatifloxacin Formulation by Proposed Methods

<table>
<thead>
<tr>
<th>Formulations</th>
<th>Label claim (mg)</th>
<th>Amount estimated*(mg)</th>
<th>% Recovery*</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Method A</td>
<td>Method B</td>
</tr>
<tr>
<td>Tablet 1</td>
<td>200</td>
<td>198±0.04</td>
<td>201±0.05</td>
</tr>
<tr>
<td>Tablet 2</td>
<td>200</td>
<td>198±0.07</td>
<td>201±0.08</td>
</tr>
</tbody>
</table>

*Values are Mean±SEM of five determinations. Tablet 1 is Gatiquin 200 mg tablets from Cipla, Ahmedabad and Tablet 2 is Gaity 200 mg tablets from Dr. Reddy’s Labs, Hyderabad.
Method for the Simultaneous Determination of Atorvastatin and Amlodipine in Tablet Dosage Form


Shri Vishnu College of Pharmacy, Vishnupur, Bhimavaram-534 202, University College of Pharmaceutical Sciences, Andhra University, Visakhapatnam-530 003, India.

A simple, precise, accurate, and rapid HPLC method has been developed, and validated for the determination of atorvastatin and amlodipine simultaneously, in combined tablet dosage form. The mobile phase used was a mixture of acetonitrile and 0.03M phosphate buffer pH 2.9 (55:45% v/v). The detection of atorvastatin and amlodipine was carried out on dual absorbance detector at 240 nm and 362 nm, respectively. Results of the analysis were validated statistically, and by recovery studies. The proposed method can be successfully used to determine the drug contents of marketed formulation.

Atorvastatin calcium, chemically, 1H-pyrrole-1-heptanoic acid, [R-(R*,R*)]-2-(4-flurophenyl)-β,δ-dihydroxy-5-(1-methylethyl)-3-phenyl-4-[(phenylamino) carbonyl]-calcium salt (2:1), is an antihyperlipoproteinemic drug, used for treatment of hypercholesterolemia. Amlodipine, chemically, 2-[(2-Aminomethoxy) methyl]-4-(2-chlorophenyl)-1,4-dihydro-6-methyl-3, 5-pyridine dicarboxylic acid, 3-ethyl-5-methyl ester, is a calcium channel antagonist, used as an anti-hypertensive drug.

Literature survey reveals that analytical methods, including Capillary zone electrophoresis, and HPLC methods, are available for the determination of atorvastatin in pharmaceutical dosage forms. The combination of atorvastatin (ATS) and amlodipine (AML) has recently been introduced into the market. This combination of amlodipine and atorvastatin can be safely used in the treatment of patients with concomitant hypertension and dyslipidemia. However, so far, no method was reported for the simultaneous estimation of atorvastatin and amlodipine, in combination.

Separation was carried out on an isocratic HPLC system (Waters), with Waters 1525 binary HPLC pump, Waters 2487 UV dual λ absorbance detector, Waters Breeze software, and RP-C18 column (150x4.6 mm I.D.; particle size 5 µm). The chromatographic estimation was performed using the following conditions: the mobile phase used was acetonitrile and 0.03 M phosphate buffer.

ACKNOWLEDGEMENTS

The authors are grateful to Dr. R. Shivakumar, Managing Director, S.R.M. Group of Educational Institutions for providing necessary facilities.

REFERENCES