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NOTE

Spectrophotometric Determination of Gemifloxacin in Pharmaceutical Formulations

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Two simple and sensitive spectrophotometric methods (method **A** and method **B**) in the visible region have been developed for the determination of gemifloxacin in bulk and in pharmaceutical formulations. Method **A** is based on the reduction of ferric chloride by gemifloxacin and subsequent complexation of ferrous ions with 2,2'-bipyridine reagent to form a red coloured chromogen with an absorption maximum of 520 nm. Method **B** is based on the reaction of gemifloxacin with ferric alum reagent under acidic conditions to form a yellow coloured product having an absorption maximum of 460 nm. The colour obeyed Beer's law in the concentration range of 6.4-32.0 and 5.1-25.6 µg/mL for method **A** and **B**, respectively. When pharmaceutical preparations (tablets) containing gemifloxacin were analyzed, the results obtained by the proposed methods are in good agreement with the labelled amounts. Recovery in both the methods is 98-101 %.

Key Words: Spectrophotometry, Gemifloxacin.

Gemifloxacin[1,2] is a recently developed third generation fluoroquinolone drug which is extremely useful in treating many infections. It has broad spectrum of activity against gram positive and gram negative organisms. Chemically, gemifloxacin is 7-[3-(aminomethyl)-4-(methoxyimino)-1-pyrrolidinyl]-1-cyclopropyl-6-fluoro-1,4-dihydro-4-oxo-1,8-naphthyridine-3-carboxylic acid and not yet official in any pharmacopoeia. Only HPLC[3] methods are reported earlier for the determination of gemifloxacin in biological fluids and in dosage forms. Gemifloxacin was found to form a red coloured chromogen with 2,2'-bipyridine reagent[4-7] and a yellow coloured chromogen with ferric alum reagent[8-10]. These reactions have not been studied earlier for its quantitative estimation. The authors have made an attempt in developing two visible spectrophotometric methods by utilizing the above reactions.

Standard solutions: The standard solutions of gemifloxacin prepared at a concentration of 51.2 µg/mL in 0.1 M HCl for method **A** and of concentration 64 µg/mL in 0.1 M HCl for method **B**.

2-2-Bipyridine in 0.1 HCl solution (0.01 M); ferric chloride (0.003 M, freshly prepared); phosphoric acid (0.2 M); ferric alum (0.1 % w/v) in 3 % nitric acid.

An ELICO UV-VIS spectrophotometer model SL-150 with 1 cm matched Quartz cells was employed for optical measurements.

Standard curve

Method A: Aliquots of standard gemifloxacin solution 1-5 mL were transferred to a series of graduated 10 mL volumetric flasks. 1 mL of 2,2'-bipyridine (0.01 M), 1 mL of ferric chloride reagent (0.003 M) were added to each flask and the flasks were heated for 0.5 h at 100 °C, cooled to room temperature. Then 2 mL of orthophosphoric acid was added to each flask. The volume was adjusted to 10 mL with distilled water. The red colour developed was measured at 520 nm against the reagent blank and a standard calibration curve was prepared.

Method B: A standard solution of 64 µg/mL was prepared. Into a series of graduated 10 mL volumetric flasks, aliquots of gemifloxacin ranging from 1-5 mL were transferred. To each tube 4 mL of ferric alum reagent was added. After 5 min, the volume was adjusted to 10 mL with distilled water and the absorbance of the yellow coloured species was measured at 460 nm against the reagent blank.

Estimation in pharmaceutical dosage forms: 20 Tablets were weighed and powdered. A quantity of the powder equivalent to 320 mg of gemifloxacin was transferred into 250 mL volumetric flask. To this 0.1 M HCl is added to dissolve the drug. The solution was then filtered and the filtrate was brought to 250 mL with 0.1 M HCl. From this 5 mL is taken and dissolved in 100 mL of 0.1 M HCl *i.e.*, it gives the concentration of 64 µg/mL. From the filtrate 40 mL is taken and dissolved in 100 mL of 0.1 HCl *i.e.*, it gives the concentration of 51.2 µg/mL after 1 in 10 dilution. From these stock solutions aliquots of range 1-5 mL are taken and further analysis was carried out as described under standard curve.

The above dosage forms are also analyzed by UV-spectrophotometric method by extraction of the drug into 0.1 M HCl and measuring the absorbance at 295 nm after suitable dilution.

Recovery were performed in both the methods by adding a known amount of drug to previously analyzed pharmaceutical preparations and also to various excipients used in formulations.

Optical characteristics such as Beer's law limits, molar absorptivity, Sandell's sensitivity and precision, accuracy of the proposed methods for gemifloxacin were given in Table-1.

TABLE-1
OPTICAL CHARACTERISTICS, PRECISION AND ACCURACY OF THE PROPOSED METHODS OF GEMIFLOXACIN

Parameter	Method **A**	Method **B**
λ_{max} (nm)	520	460
Beer's law limit (µg/mL)	5.1-25.6	6.4-51.2
Sandell's sensitivity (µg/cm^2/0.001 abs.unit)	0.005	0.007
Molar absorptivity (L mol^{-1} cm^{-1})	2.18×10^3	0.550×10^3
Regression equation*		
Intercept (a)	0.0020	0.0020
Slope (b)	0.0011	0.0014
Correlation Coefficient (r)	0.9998	0.9997
% RSD**	1.85	1.75

*Y = A + bx, where X is the concentration of GEF in µg mL^{-1} and Y is the absorbance of respective λ_{max}; **for six determination.

The result on the analysis of pharmaceutical formulations by the proposed and reference methods are in good agreement (Table-2). The additives usually present in the pharmaceutical formulations do not interfere. The results indicate that the proposed methods for the estimation of gemifloxacin are highly sensitive and can be used for the routine determination of gemifloxacin in bulk and in pharmaceutical formulations.The proposed method is rapid,accurate and reproducible. The blood red coloured complex resulting from gemifloxacin with 2,2'-bipyridine and iron(III).

TABLE-2
ANALYSIS OF PHARMACEUTICAL FORMULATIONS USING PROPOSED AND REFERENCE METHODS

Pharmaceutical formulation	Labelled amount (mg/tablet)	Amount found by			Recovery (%)
		Method **A**	Method **B**	Reference method* (mg)	
Table I	320	319.85	319.92	319.84	99.95
Table II	320	319.73	319.86	319.97	100.10
Table III	320	319.82	320.13	318.93	99.89

* UV Method.

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