

Spectrophotometric method for the determination of Gemifloxacin mesylate in bulk and Pharmaceutical formulations

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ABSTRACT

A simple, sensitive and reproducible spectrophotometric method was developed for the determination of Gemifloxacin mesylate in bulk and its pharmaceutical formulations. This method was based on the reaction of Gemifloxacin mesylate with Fe(III) of ferric nitrate and 0.1M hydrochloric acid to produce a orange colored chromogen (λ_{\max} at 471nm). Results of analysis were validated statistically and by recovery studies. This method was successfully employed for the determination of Gemifloxacin mesylate in various pharmaceutical preparations.

Key words: Gemifloxacin mesylate, Visible spectrophotometric determination, Beer's Law.

Gemifloxacin mesylate is a third group of fluoroquinolone active against *s.pneumonia*. It is useful in treatment of chronic bronchitis and mild to moderate pneumonia. It is chemically, (R,S)-7-[4z-3-(amino methyl)-4-(methoxy imino)-1-pyrrolidinyl]-1-cyclopropyl-6-fluoro-1, 4dihydro-4-oxo-1, 8-naphthyridine-3-carboxylic acid¹. A through survey of literature revealed only LCMS was reported for the determination of Gemifloxacin mesylate in human plasma². The analytically useful function groups of Gemifloxacin mesylate have not been fully exploited. The authors have made some attempts in this direction and succeeded in developing this spectrophotometric method for the assay. Method is based on reaction of Gemifloxacin mesylate with Fe³⁺ and 0.1 M hydrochloric acid to produce orange chromogen (λ_{\max} at 471nm).

Instrumentation

Spectral and absorbance measurements were made with Shimadzu UV-Visible Double beam spectrophotometer model-1700 with pair of 10 mm quartz cell.

Reagents

All the chemicals used were of analytical grade. Pure raw material of Gemifloxacin mesylate was obtained as a gift sample from Orchid

Pharmaceuticals, Chennai. The formulations used were purchased from local pharmacy. All the solutions were freshly prepared with double distilled water. Freshly prepared solutions were always used 1%w/v ferric nitrate reagent was prepared by dissolving 1gm of ferric nitrate in 100ml of 3M nitric acid.

Standard and Sample solution of Gemifloxacin mesylate

About 100mg of Gemifloxacin mesylate (bulk and formulations) was accurately weighed and dissolved in 100ml of 0.1M hydrochloric acid in a volumetric flask to make a solution of 1 mg/ml standard solution and further dilutions are made with the same solvent.

Assay procedure

Aliquots 1 to 5 ml of standard Gemifloxacin mesylate (100 μ g/ml) was transferred to a series of 10ml volumetric flask. Each 10ml volumetric flask, added 0.6ml of 1%w/v ferric nitrate reagent solution and made up to the mark with 0.1M hydrochloric acid and kept aside for few minutes. The absorbance of the orange colour chromogen was measured at 471nm against the reagent blank. The amount of Gemifloxacin mesylate was computed from the calibration curve.

The proposed method was based on redox reaction in which Gemifloxacin mesylate is oxidized by ferric nitrate. The same method was already reported for the gatifloxacin, which is an analogue of the our drug. The optical characteristics such as absorption maxima, beer's law limit, molar absorptivity and sandell's sensitivity for this method were presented in Table 1. The regression analysis using the method of least squares was made for the slope (a), intercept (b) and correlation coefficient (r) obtained from different concentrations was summarized in Table 1.

The accuracy of this method was ascertained by comparing the results obtained with the proposed method in the case of formulations and is presented in Table 2. As an additional check on the accuracy of this method, adding known amounts of pure drug to pre-analyzed formulations. Performed recovery experiment and percent recovery values obtained are listed in Table 2.

Table 1: Optical characteristics of Gemifloxacin mesylate

Parameters	Results
λ_{\max} (nm)	471
Beers law limit ($\mu\text{g/ml}$)	10-50
Sandell's sensitivity ($\mu\text{g/cm}^2/0.001 \text{ A.U}$)	0.246913
Molar extinction coefficient ($\text{L mol}^{-1} \text{ cm}^{-1}$)	1348.95
Correlation coefficient (r)	0.9999
Regression equation* ($y=mx+c$)	$Y=0.00405x+0.0029$
Slope(m)	0.00405
Intercept(c)	0.0029
LOD ($\mu\text{g/ml}$)	0.45522
LOQ ($\mu\text{g/ml}$)	1.3794
Standard error	0.0004830

* $Y = mx+c$, where 'Y' is the absorbance and c is the concentration of Gemifloxacin mesylate in mg/ml ** For six replicates

Table 2: Assay and recovery studies of Gemifloxacin mesylate in Pharmaceutical formulations

Formulations	Labeled amount mg/tablet	*Amount found (mg) \pm SD	% Recovery by proposed method
Gemistar	320 mg	319.16 \pm 0.90	98.69
Gembax	320 mg	319.61 \pm 0.64	99.84
G-cin	320 mg	319.11 \pm 0.74	99.90

* mean of three estimation at each levels

Recovery experiment indicated the absence of interferences from the commonly encountered pharmaceutical additives and excipients.

Thus, the proposed method is simple and sensitive with reasonable precision and accuracy. This can be used for the routine determination of Gemifloxacin mesylate in quality control analysis.

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