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NOTE

Reverse Phase High Performance Liquid Chromatographic Determination of Gatifloxacin in Gatifloxacin Eye Drops

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A simple and precise reverse phase HPLC method was developed for the estimation of gatifloxacin in ophthalmic formulations. The method was carried out on a Thermoquest C8 (15 cm × 4.6 mm i.d., 5 μ) column with mobile phase consisting of buffer monobasic potassium phosphate 4.0827 g diluted to 1 L in the proportion Buffer : acetonitrile : triethylamine (650 : 350 : 2) mixed properly and 2 g of sodium lauryl sulphate was added. pH was adjusted to 3.0 with orthophosphoric acid at a flow rate 1.5 mL/min. Detection was carried out at 290 nm. The retention time of gatifloxacin was 10.9. The validation of the proposed method was also carried out.

Key words: Gatifloxacin, HPLC reverse phase liquid chromatography.

Gatifloxacin is an antibacterial agent, a synthetic broad-spectrum 8-methoxy-fluoroquinolone antibacterial agent for oral or intravenous administration. Chemically, gatifloxacin is (±)-1-cyclopropyl-6-filuoro-1,4-dihydro-8-methoxy-7-(3-methyl-1-piperazinyl)-4-oxo-3-quinolinecarboxylic acid sesquihydrate. There are several methods[1–10] for the determination of gatifloxacin. There is no reporton HPLC determination of gatifloxacin. The aim of this work was to develop a simple, rapid HPLC method for the determination of gatifloxacin in pharmaceutical dosage form.

Orthophosphoric acid AR grade and acetonitrile of HPLC grade, monobasic potassium phosphate buffer AR grade, sodium lauryl sulphate AR grade and triethylamine AR grade, supplied by Qualigen Fine Chemicals. Water PLC grade obtained from Elga Purification System was used. Reference standard of gatifloxacin was used.

A Merck-Hitachi HPLC system was used for the analysis. The method used was carried out on a Merck C8 (15 cm × 4.6 mm i.d. 5 μ) column as stationary phase and buffer : acetonitrile : triethylamine (650 : 350 : 2) mixed properly and 2 g of sodium lauryl sulphate was added. pH was adjusted to 3.0 with orthophosphoric acid as a mobile phase at a flow rate 1.5 mL/min. A rheodyne injector with 20 μL loop was used for the injection of sample. Detection was done at 290 nm. The mobile phase was filtered through a 0.45 μ membrane filter and degassed. The separation was carried out at room temperature of about 25°C.

Preparation of standard solution

60 mg of gatifloxacin working standard was accurately weighed in 50 mL

volumetric flask and made up to the volume with mobile phase. Further, 5 mL of this solution was diluted to 100 mL with mobile phase.

Accurately measured 2 mL of the sample was transferred to a 100 mL volumetric flask, diluted with mobile phase to volume, mixed and used for analysis.

Procedure

20 μL of standard and sample solution were injected into the injector of a liquid chromatograph. From the peak ratio of area of gatifloxacin standard and sample was completed. The values are given in Table-1

TABLE-1

Sample area	Standard area	Content (%)	RSD (%)
5693028	5346979	98.90	
5715269	5321422	99.30	
5701248	5362718	99.03	0.164
5714612	5355915	99.23	
5699421	5331181	99.03	

Recovery

To study the accuracy, reproducibility and precision of the proposed method, recovery experiments were carried out. A fixed amount of pre-analyzed sample was taken and standard was added at three different levels in placebo.

Linearity

The plot of ratio of area of gatifloxacin *vs.* respective concentrations of drug are found to be linear in the range of 0.03, 0.06, 0.09, 0.12 and 0.15 mg/mL respectively with correlation coefficient r = 0.999229372.

Chromatography

The optimum mobile phase buffer : acetonitrile : triethylamine (650 : 350 : 2) is selected because it is found to be ideally resolved. The peak of gatifloxacin 10.9 without interference of placebo as the retention time is shown in Fig. 1.

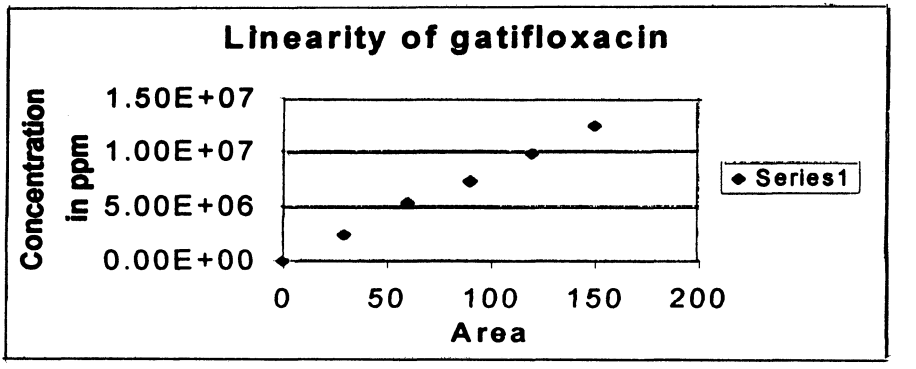

Fig. 1

Wavelength is selected by scanning standard over a wide range of wavelengths 200–400 nm. Gatifloxacin showed reasonable absorbance at 290 nm.

The contents of gatifloxacin found by proposed method are shown in Table-1. The low value of RSD indicates that the method is precise and accurate. The mean recoveries of gatifloxacin are 100.45, 98.99 and 99.70% as shown in Table-2.

TABLE-2

Sr. No.	Concentration in ppm	Standard area	Sample area	% Recovery	Coefficient of variation	RSD
1st level	48	3946399	3964257	100.45	0.99221467	0.008552
2nd level	60	5346979	5293208	98.99	0.99956182	0.001685
3rd level	72	6091507	6073530	99.70	0.99991295	0.009526

Conclusion

The proposed method gives good separation and within short analysis time (< 15 min). The method is very simple, rapid and does not involves use of complex instruments. High percentage of recovery shows that the method is free from interference of the excipients used in the formulations. Therefore the method can be useful in routine quality control analysis of this drug.

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