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NOTE

Development and Validation of High Performance Liquid Chromatography Method for Analysis of Gatifloxacin and Its Impurity

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A simple isocratic reverse phase high performance liquid chromatographic method was developed for the intermediates and finished products in reaction stream during process development of gatifloxacin. The method was utilized successfully in analyzing the reaction streams, related substances in final product and for the assay in drug.

Key Words: Gatifloxacin, RP-HPLC, Methyl piperazine.

Gatifloxacin level in biological fluids, different pharmaceutical formulations and as a raw material for related substances have previously been determined by spectrophotometric, gas chromatographic techniques[1-3]. No method is reported for the analysis of reaction mixture obtained in the preparation of gatifloxacin in the literature.

The HPLC system consisted of Jasco make UV/Vis detector model 1575, along with Borwin software (Integrator) were used. Analysis were performed on stainless steel column containing C-18 packing, 5 μ ODS (25 cm × 4.6 mm).

Accurately weighed 100 mg (± 5 mg) of working standard of gatifloxacin and transferred to a 50 mL volumetric flask. Add 10 mL of mobile phase and sonicated until dissolved. Allow to cool at room temperature and diluted it further with mobile phase. Accurately weighed 5 mg of the working standard of 2-methyl piperazine and transferred to 50 mL volumetric flask. Transferred 4 mL of solution 2-methyl piperazine in gatifloxacin solution in volumetric flask and diluted it further with mobile phase. The sample solution was also made on the same procedure.

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System suitability data (Table-1) showed that method is accurate. As per USP XXVII, system suitability was carried out in freshly prepared reference solution B to check various parameters such as efficiency, resolution and peak tailing.

TABLE-1

Compound	Standard deviation	RSD	Theoretical plates	Resolution factor	Tailing factor
2-Methyl piperazine	5.04619	0.6621	5370	1.6	1.1
Gatifloxacin	302.6325	1.624284	7271	–	–

The reproducibility is more than 99 % for the product as impurity on analysis of three consecutive days (Table-2).

TABLE-2

Gatifloxacin		2-Methyl piperazine	
Weight (mg/mL)	Recovery (%)	Weight (mg/mL)	Recovery (%)
1	99.32	0.01	98.73
1	98.84	0.01	99.12
1	99.50	0.01	99.36
Average (%)	99.38	Average (%)	99.06
SD	0.2844732	SD	0.29580399
RSD	0.2862479	RSD	0.29861093
CV	0.5724959	CV	0.59722186

TABLE-3

Gatifloxacin			
Concentration (mg/mL)	Actual concentration taken (mg/mL)	Actual concentration recover (mg/mL)	Recovery (%)
1.0	1	0.9778	0.9778
	1	0.9847	0.9847
	1	0.9846	0.9846
	1	1.0031	1.0031
X = 0.98755; S = 0.01086; CV = 1.09949; 100 % of theoretical concentration 95 % confidence limits = 0.98755; 0.01726426			
2-Methyl piperazine			
0.01	0.0100	0.00993	0.9925
	0.0100	0.00995	0.9946
	0.0100	0.00991	0.9905
	0.0096	0.00966	1.00614583
X = 0.99594; S = 0.00701; CV = 0.70377; 100 % of theoretical concentration 95 % confidence limits = 0.99594; 0.01114444			

Table-3 showed that this method has recovery more than 99 % of all the impurities along with the product (gatifloxacin), which is considered to be excellent during analysis of product and impurity. Ruggedness of the method is also shown in Table-3. The mean recoveries of impurity were in the range of 99.3 to 100.0 %, which confirms that there is no interference from the mobile phase, thus further confirm the reproducibility and reliability of the method.

The linearity curve (slope) and regression data for the product and its impurity, confirmed that the method is accurate and reproducible (Table-4). The lower values of reproducibility indicate that the method is precise and accurate.

TABLE-4

Criteria measured	Gatifloxacin		2-Methyl piperazine	
	AA	Results	AA	Results
Concentration range (%)	40-150	–	40-150	–
Graphic plot (Points)	24		24	
R		0.998370		0.99994
R^2		0.996743		0.99988
Average fractional recovery (%)	99-101	100.06	95-105	99.20
Slope (A *vs.* B)	1.00	1.01	1.00	0.989
Coefficient variation	–	0.7730688	–	0.72090

AA = Drug substance acceptance value.

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