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Determination of Fexofenadine Hydrochloride in Pharmaceutical Dosage Form By Reverse Phase High Performance Liquid Chromatography Method

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ABSTRACT

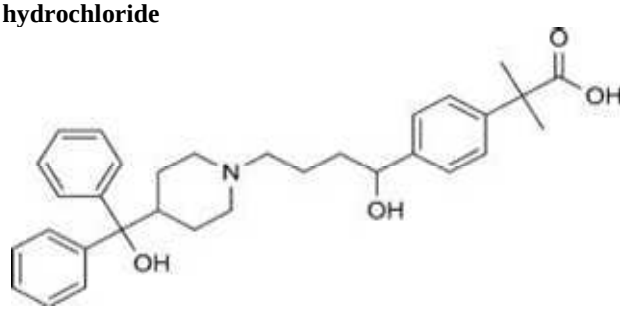
Rapid and accurate reverse phase high performance liquid chromatography method is described for determination of fexofenadine hydrochloride from the pharmaceutical dosage form. It was observed that Polaris C18 (15 x 4.6 mm i.d.) with 5 µ particle size column showed most favorable chromatographic pattern over the other columns. The mobile phase consisted of buffer and acetonitrile (65:35 % v/v). The detection was carried out at wavelength 220 nm. The method was validated for system suitability, linearity, accuracy, precision, robustness and stability of sample solution with the linear range 5-15 µg/ ml. The method has been successfully used to assay of pharmaceutical dosage form i.e. tablets with good recoveries.

Key words: Fexofenadine hydrochloride, potassium dihydrogen phosphate , acetonitrile, HPLC.

INTRODUCTION

Fexofenadine is described as second or third generation antihistamine. Its chemical name is RS -2 [4-(hydroxydiphenyl- methyl)-1 piperidyl]butyl] phenyl]- 2methyl-propanoic acid. ($C_{32}H_{39}NO_4$). It is indicated for relief from physical symptoms associated with seasonal allergic rhinitis and for the treatment of chronic urticaria. It prevents the aggravation of rhinitis and urticaria and reduces the severity of the symptoms associated with those conditions, providing relief from the repeated sneezing, runny nose, itchy eyes and generated body fatigue. This drug is official in USP [1], IP[2] pharmacopoeia. In literature survey EE capillary electrophoresis [3], HPLC [4-6] and spectrophotometric [7-10], non aqueous titration [11] methods have been reported for assay of fexofenadine hydrochloride.

Structure of fexofenadine hydrochloride



MATERIALS AND METHODS

Chemical and reagents

Reference standard of fexofenadine hydrochloride was obtained from reputed firm with certificate of analysis. Potassium dihydrogen phosphate, acetonitrile and ortho-phosphoric acid were used of analytical grade and HPLC grade water was used from Millipore.

Instrumentation

The HPLC system used was MERCK Hitachi HPLC system equipped with auto sampler (D 7200 separation module) and UV detector (D- 7400). The chromatogram was recorded and peaks quantified by means of PC based EZChrom Elite software.

A SHIMADZU analytical balance (0.01 mg) was used.

Preparation of Standard preparation

Standard solution

A 10 mg of standard fexofenadine hydrochloride was weighted accurately and transferred in 100 ml volumetric flask. About 50 ml of diluent was added and sonicated for 5 minutes. The volume was adjusted up to the mark with diluent to give concentration as 100 µg /ml. The diluent was 8.6 g. of potassium dihydrogen phosphate in 1000 ml of HPLC grade water and pH 4 was adjusted with ortho- phosphoric acid and acetonitrile in ratio as 65:35 % (v/v).

Sample preparation

Twenty tablets were weighed accurately and average weight of each tablet was determined. About 10 mg of fexofenadine hydrochloride sample was weighted accurately and transferred in 100 ml volumetric flask. About 50 ml of diluent was added and sonicated for 10 minutes. The volume was adjusted up to the mark with diluent to give concentration as 100 µg /ml.

Chromatographic condition

Chromatographic separation was performed at ambient temperature on a reverse phase Polaris C18 (15 x 4.6 mm i.d.) with 5 µ particle size column. The mobile phase was a mixture of buffer and acetonitrile (65:35 % v/v). The buffer was mixtures of 8.6 g of potassium dihydrogen phosphate solution adjusted the pH 4 with ortho-phosphoric acid. The flow rate of the mobile phase was adjusted to 1 ml /min. The detection was carried out at wavelength 220 nm. (Fig. no.1) The injection volume of the standard and sample solution was set at 20.0 µl.

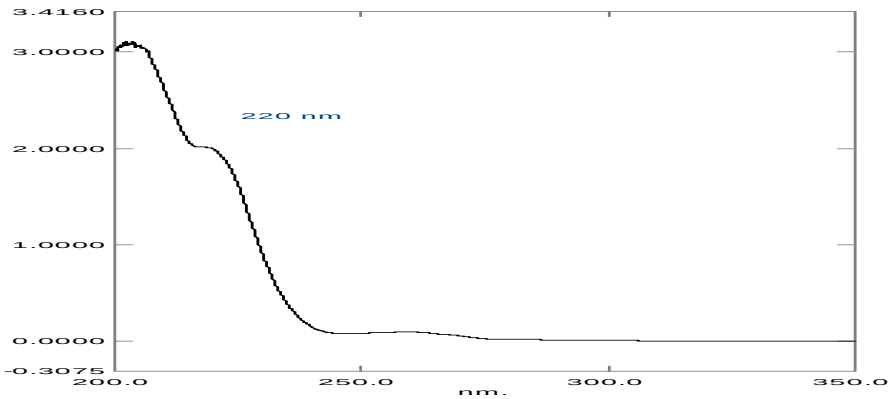


Figure 1: UV spectra of fexofenadine hydrochloride

Method validation

System suitability

System performances of developed HPLC method were determined by injecting standard solutions. Parameter such as theoretical plates (N), symmetry, area and % area were determined. The results are shown in table 1 which indicates good performance of the system.

Table 1: System suitability parameters evaluated on standard solution of fexofenadine hydrochloride

Retention Time	Area	Area %	Asymmetry
4.1	1374666	100	1.173

Specificity

Specificity is the ability of the method to resolve the active ingredients. Hence blank, standard fexofenadine hydrochloride was injected to prove specificity. The typical chromatogram of the standard and sample assayed are given in figure 2 and 3 respectively.

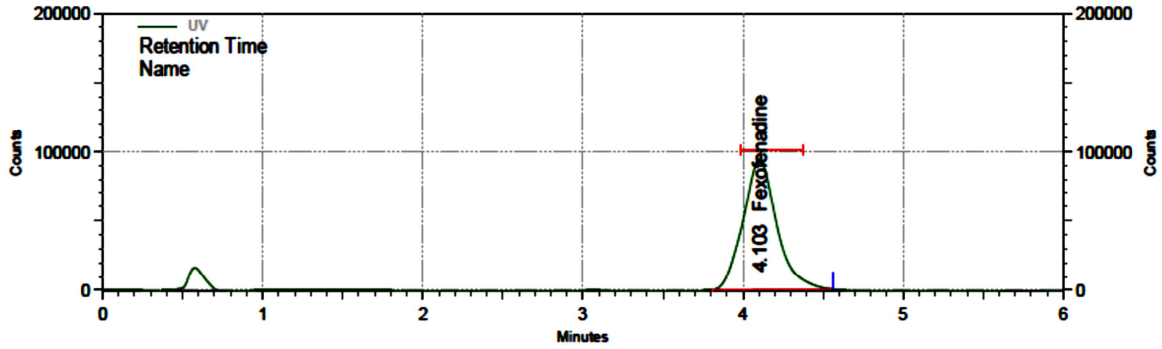


Figure 2: Chromatogram of fexofenadine hydrochloride (standard)

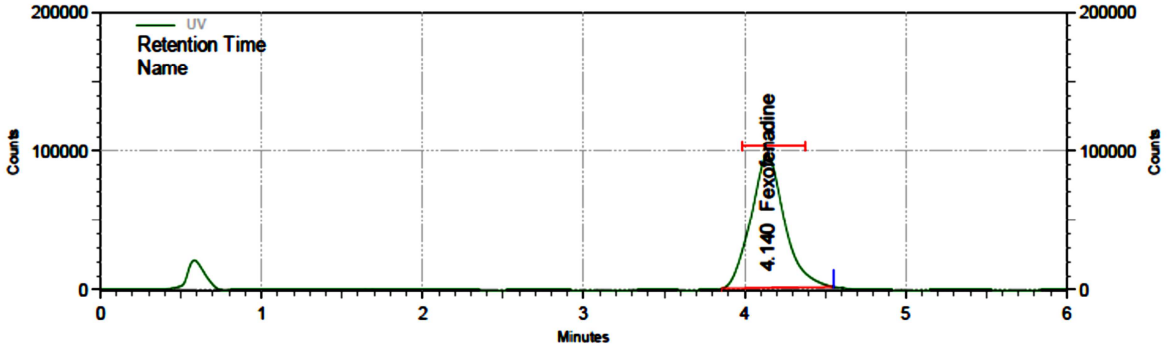


Figure 3: Chromatogram of fexofenadine hydrochloride(sample)

Linearity

Under the experimental conditions described above, linear calibration curve were obtained throughout the concentration range studied. Regression analysis was done on the peak area (y) v/s concentration (x). The regression analysis data obtained is tabulated in table no. 2.

Table 2: Statistical evaluation of the data subjected to regression analysis

Parameters	fexofenadine hydrochloride
Correlation Coefficient (r)	0.9999
% Intercept (y)	-7961.4
Slope (m)	123539

Accuracy

The accuracy method was determined by applying proposed method to synthetic mixture containing known amount of drug corresponding to 80 %, 100 % and 120 %. The accuracy was then calculated as the percentage of analyte recovered by the assay. The results of the recovery analysis are enclosed under table no.3.

Table 3: Statistical evaluation of the data subjected to accuracy of fexofenadine hydrochloride

level	Test no.	Weight in mg	Area	Quantity added in µg /ml	Quantity recovered in µg /ml	% recovery	mean recovery
80%	1	1.11	1098835	8.96	8.94	99.81	**99.54**
	2	1.16	1091337	8.96	8.88	99.13	
	3	1.12	1097591	8.96	8.93	99.69	
100%	1	1.15	1375277	11.2	11.19	99.93	**99.81**
	2	1.10	1374158	11.2	11.18	99.85	
	3	1.13	1371137	11.2	11.16	99.63	
150%	1	1.20	1656125	13.44	13.48	100.28	**100.24**
	2	1.09	1657741	13.44	13.49	100.38	
	3	1.16	1652340	13.44	13.45	100.06	
					Mean recovery of all level		**99.86**

Precision

The method precision was established by carrying out the analysis of fexofenadine hydrochloride. The assay was carried out of the drug using analytical method in five replicates. The value of relative standard deviation lies well with the limits. The results of the same are tabulated in the table no. 4.

Table 4: Statistical evaluation of the data subjected to method precision of fexofenadine hydrochloride

Test	Weight of test	Area	% assay
Solution-1	1.12	1374666	99.89
Solution-2	1.1	1376483	98.24
Solution-3	1.11	1378210	99.25
Solution-4	1.09	1379446	99.33
Solution-5	1.05	1377330	98.21
Solution-6	1.07	1375040	98.99
	Mean Assay		**98.98**
	SD		**0.658**
	RSD		**0.665**

Robustness

The robustness of the method was determined to check the reliability of an analysis with respect to deliberate variations in method parameters.

The typical variations are given below:
Variation in the flow rate by $\pm$ 0.2 ml /min
Variation in mobile phase composition by $\pm$ 2 %
Variation in wavelength ± 5 nm

The results of the analysis of the samples under the conditions of the above variation indicated the nature of robustness of the method.

Method application

A sample equivalent to 10 mg of fexofenadine hydrochloride sample was weighted accurately and transferred in 100 ml volumetric flask. About 50 ml diluent was added and sonicated for 10 minutes to dissolve it. Further volume was made up to the mark with the diluent to give 100 µg /ml. From this solution 20 µl was injected specific conditions. The analyte peak was identified by comparison with that of respective standard. The (%) assay results were expressed in table no. 4. It indicates the amount of fexofenadine hydrochloride in the product meets the requirement.

RESULTS AND CONCLUSION

The reproducibility, repeatability and accuracy of the proposed method were found to be satisfactory which is evidenced by low values of standard deviation and percent relative standard deviation. The accuracy and reproducibility of the proposed method was confirmed by recovery experiments, performed by adding known amount of the drug to the pre-analyzed active pharmaceutical ingredient and reanalyzing the mixture by proposed method. Thus the proposed RP-HPLC method is used for estimation of fexofenadine hydrochloride from active pharmaceutical ingredient. It is more precise, accurate, linear, robust, simple and rapid method. Hence the proposed

RP-HPLC method is strongly recommended for the quality control of the raw material, active pharmaceutical ingredient and pharmaceutical formulation.

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