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Assay of fexofenadine hydrochloride in pharmaceutical preparation by visible spectrophotometry

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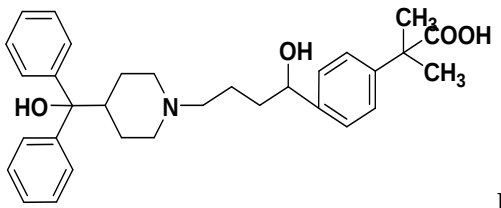
ABSTRACT

A simple and sensitive visible spectrophotometric method is described for the determination of fexofenadine hydrochloride in bulk and solid dosage forms. This method is based on the reaction of drug with aromatic aldehyde such as Vanillin in the presence of sulphuric acid in non-aqueous medium and formed coloured condensation product with an absorption maximum of 590nm for the Method. The Beers law obeyed in the concentration range of 50-300 µg/ml. The proposed method is validated with respect to accuracy, precision, linearity and limit of detection. The suggested procedure is successfully applied to the determination of the durg in pharmaceutical preparation, with high percentage of recovery, good accuracy and precision. The result of analysis is validated satistically by repeatability and recovery studies. The result is found satisfactory and reproducible. This method is applied successfullyl for the estimation of fexofenadine hydrochloride in tablet dosage form without the interference of excipients.

Keywords: Analysis, Beer's law, condensation reactions, Vanllin, Tablets.

INTRODUCTION

Fexofenadine HCl (FFH), chemically designated as (±)-4-[1-hydroxy-4-(4- hydroxydiphenylmethyl)-1- piperidinyl]-butyl]-∝,∝-dimethyl benzeneaceticacid hydrochloride is a histamine H1 receptor antagonist used in patients with allergic rhinitis.The molecular weight is 538.13 and the empirical formula is C32H39NO4•HCl.FFH can be estimated by HPLC methods but no spectrophotometric method was reported in literature till date. Hence an attempt has been made to develop and validate a simple, economic rapid and accurate method. Some analytical methods which include HPLC, LC-MS and visible spectrophometric have been reported in the literature or the determination of FFH in pharmaceutical preparations. The main purpose of the present study was to establish a relatively simple, sensitive and validated visible spectrophotometric method for the determination of FFH in pure form and in pharmaceutical dosage forms, since most of the previous methods have been found to be relatively complicated and tedious. The proposed method Vanllin, in presence of sulphuric acid in non aqueous medium and colored condensation products are formed and stable for 30 minutes. This method can be extended for the routine assay of FFH formulations.



Hcl

Fig.1: chemical structure of fexofenadine hydrochloride

MATERIALS AND METHODS

Apparatus and chemicals

A Shimadzu UV-Visible spectrophotometer 1601 with 1cm matched quartz cells was used for all spe4ctral measurements. All the chemicals use4d were of analytical grade Tablets were purchased from local market. Sulphuricacid (14M). Vanillin (BDH, 0.4%, W/V 2.63X 10^{-2}M); in methanol was prepared.

Preparation of standard drug stock solution

The stock solution of drug was prepared by dissolving 100 mg in 100 ml distilled water. A portion of this stock solution was diluted stepwise with the distilled water to obtain the working standard drug solution of concentrations of 100 µg/ml. from the stock solution, a series of standards were freshly prepared during the analysis day.

Preparation of sample solution.

Twenty tablets were weighed and finely powdered. A quantity of tablet powder equivalent to 100 mg of FFH taken in volumetric flask (100 ml) was shaken with methanol (10.0 ml) for 10 min and the volume was made upto the mark with distilled water. The solution was then filtered through whatman filter paper and the aliquot portion of the filtrate was diluted to 100.0 ml with distilled water to get sample solution.

Assay:

To each of 10ml calibrated tubes, aliquots (0.5 – 2.5 ml, 500µg/ml) of standard FFH solution, 1.0 ml of vanillin and 3.0 ml of conc. Sulphuric acid were added successively and the total volume in each flask was brought to 9 m by the addition of methanol and placed in a heating water bath for 15 min. then the flasks were cooled and made up to the mark with methanol and the absorbances were measured after 5 min. at 593 nm against a reagent blank prepared in a similar way. The amount of FFH present in sample solution was calculated from its calibration graph.

RESULTS AND DISCUSSION

Optimum operating condition used in the procedure were established by adopting variation of one variable at a time (OVAT) method. The effect of various parmeters such a time, volume and strength of reagents and acid solution and solvent for final dilution of the colored species were studied. The optical chracteristis such as Beer's law limits, Sandell's sensitivity, molar extinction coefficient, percent relative standard deviation (calculated from the six measurements containing 3/4 th of the amount of the upper Beer's law limits) were calculated for all the methods and the results are summarized in Table-1. Regression characteristics like standard deviation of slope (s_a), standard error of estimation (S_e), % range o error (0.05 and 0.01 confidence limits) were calculated for both the methods and are shown in Table-1.

Commercial formulations containing FFH were successfully analyzed by the proposed methods. The values obtained by the proposed and reference methods for formulations were compare statistically by the t-and F-test and found not to differ significantly. As an additional demonstration of accuracy, recovery experiments were performed by adding a fixed amount of the drug to the pre4 analyze formulations at three different concentration levels. These results are summarized in Table-2.

The ingredients usually present in formulations of FFH did not interfere with the proposed analytical methods. Among the different aldehydes tried for developing the color in acidic conditions vanillin was pre4ferred over others due to high sensitivity. This method involves the condensation drug with vanillin in acid medium. This method can be extended for the routine assay of FFH formulations.

Chemistry of colored specieis:

In the present investigation, the presence of imino group in indole moiety (secondary amine) permits the development of visible spectrophotometric methods for it determination through the4 condensation reaction with aromatic aldehydes. The formation of colored species with these reagents may be assigned through above analogy as shown in figure 2.

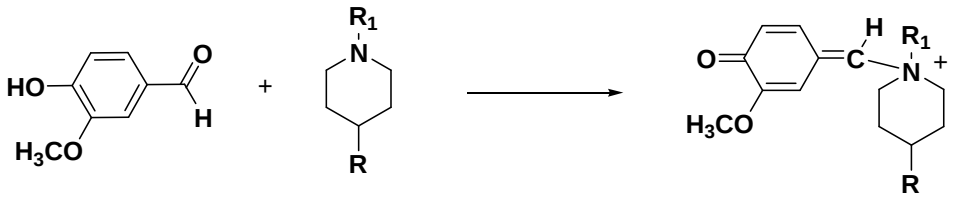


Fig.2:Probable scheme for proposed method

Table 1: optical characteristics, precision and accuracy of proposed method.

Parameters	values
λ_{max} (nm)	590
Beer's law limit (µg/ml)	50-300
Sandell's sensitivity (µg/cm^2/0.001 abs. unit	1.194
Molar absorptivity (Liter/mole/cm)	2.951x10^4
Regression equation	
(Y)*= a+bc	
Intercept(a)	0.0002
Slope (b)	0.001
%RSD	0.342
%Range of errors (95% Confidence limits)	
0.05 significance level	0.394
0.01 significance level	0.617

Y*=a+bc ; where y=absorbance. C= concentration of FFH inµg/ml.

Table 2: Analysis of FFH in pharmaceutical formulations by proposed and reference methods.

Method	Formulations	Labeled Amount(mg)	Found±SD[a]	Recovery (%)	[b]t-test	[c]f-test
Vanillin	Tablet 1	120	120.04±0.04	100.03	2.18	4.00
	Tablet 2	120	119.99±0.07	99.98	0.13	1.30

[a] *Mean±standard deviation (n=5) [µg/tablet];* [b] *Tabulated t-value at 95% confidence level is 2.31*

[c] *Tabulated F-value at 95% confidence level is 6.39; Fexofenadine Hydrochloride tablet Aventis pharma limited, Ankleshwar*

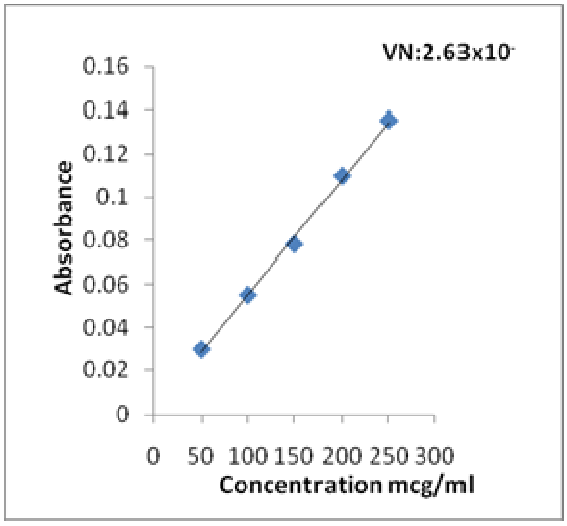


Fig. 3. Beers Law plot of FFH with VN System

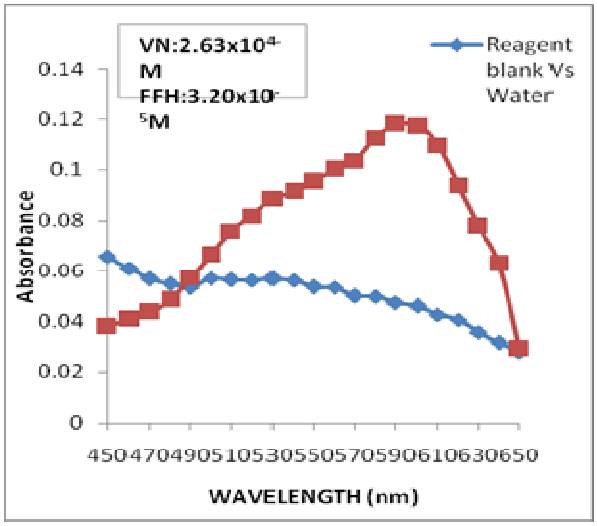


Fig. 4 Absorption spectra of FFH with VN system and its reagent blank

CONCLUSION

The proposed methods for FFH determination have many advantages over other analytical methods due to its rapidity, lower cost and environmental safety. Unlike HPLC. HPTLC procedures, the instrument is simple and is not costly. Economically, all the analytical reagents are inexpensive and available in any analytical laboratory . This method can be extended for the routine assay of FFH formulations.

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