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THE ESTIMATION OF VILDAGLIPTIN IN TABLETS BY RP-HPLC.

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ABSTRACT

A simple, precise, rapid and accurate reverse phase HPLC method developed for the estimation of Vildagliptin in Tablets dosage form. A Inertsil ODS 3V C18, 250x4.6 mm i.d, 5 µm partical size, with mobile phase consisting of Acetonitrile and 0.03 M potassium dihydrogen phosphate in water (pH 3.5 adjusted with o-phosphoric acid) in the ratio of 80:20 v/v was used. The flow rate was 1 ml/min and the effluents were monitored at 205 nm. The retention time was 3.857 min. The detector response was linear in the concentration of 50-300 mcg/ml. The respective linear regression equation being Y= 33227.348x+101075.26. The limit of detection and limit of quantification was 0.25 and 0.75 mcg/ml respectively. The percentage assay of Vildagliptin was 98.56 %. The method was validated by determining its accuracy, precision and system suitability. The results of the study showed that the proposed RP-HPLC method is simple, rapid, precise and accurate, which is useful for the routine determination of Vildagliptin in bulk drug and in its pharmaceutical dosage form.

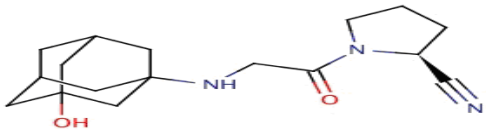
KEYWORDS: Vildagliptin, RP-HPLC, Estimation, and Tablets.

INTRODUCTION

Vildagliptin (previously identified as LAF237, trade name Galvus) is a new oral anti-hyperglycemic agent (anti-diabetic drug) of the new dipeptidy[1] peptidase-4 (DPP-4) inhibitor class of drugs. Vildagliptin inhibits the inactivation of GLP-1[2,3] and GIP[3] by DPP-4, allowing GLP-1 and GIP to potentiate the secretion of insulin in the beta cells and suppress glucagon release by the alpha cells of the islets of Langerhans in the pancreas. Vildagliptin has been shown to reduce hyperglycemia in type 2 diabetes mellitus.[2]

Literature survey reveals many Chromatographic methods for the determination of Vildagliptin in biological fluids. So far, no assay procedure has been reported for the estimation of Vildagliptin from pharmaceutical dosage forms.

The availability of an HPLC method with high sensitivity and selectivity will be very useful for the determination of Vildagliptin in pharmaceutical formulations. The aim of the study was to develop a simple, precise, and accurate reversed-phase HPLC method for the estimation of Vildagliptin in bulk drug samples and in pharmaceutical dosage form.



Chemical structure of Vildagliptin.

EXPERIMENTAL

MATERIALS AND METHODS

Vildagliptin was obtained as a gift sample from Novartis, Bangalore. Potassium dihydrogen phosphate and o-Phosphric acid were of analytical grade, and supplied by M/s S.D.Fine Chem Limited, Mumbai. Acetonitrile and water used were of HPLC grade (Qualigens). Commercially available Vildagliptin Tablet (Galvus® 50 mg Novartis) was procured from local market.

Instrument: Quantitative HPLC was performed on liquid Chromatograph, Waters separation 2996, PDA detector module equipped with automatic injector with injection volume 20 µl, and 2693 pump. An Inertsil ODS 3V C-18 column (250x4.6 mm i.d; particle size 5 µm) was used. The HPLC system was equipped with Empower Software.

HPLC Conditions: The contents of the mobile phase were Acetonitrile and 0.03 M Potassium dihydrogen phosphate in water (pH 3.5 adjusted with o-phosphoric

acid) in the ratio of 80:20 v/v was used. They were filtered before use through a 0.45 µm membrane filter, and pumped from the respective solvent reservoirs to the column at a flow rate of 1.0 ml/min. The run time was set at 10.0 min and the column temperature was ambient. Prior to the injection of the drug solution, the column was equilibrated for at least 30 min with the mobile phase flowing through the system. The eluents were monitored at 225 nm.

***Preparation of Standard Stock solution*:** A standard stock solution of the drug was prepared by dissolving 25 mg of Vildagliptin in 10 ml volumetric flask containing 5ml of diluent (50:50 v/v Acetonitrile: water), sonicated for about 15 min and then made up to 10 ml with diluent to get a 2.5 mg/ml standard stock solution.

***Working Standard solution*:** 5.0 ml of the above stock solution was taken in 50 ml volumetric flask and thereafter made up to 50 ml with diluent (50:50 v/v Acetonitrile: water) to get a concentration of 250 µg/ml.

***Preparation of Sample solution*:** Twenty tablets (Galvus® 50 mg, Novartis Pharma) were weighed, and then powdered. A sample of the Tablet powder, equivalent to 25 mg of the active ingredient, was mixed with 10 ml of diluent [50:50 v/v Acetonitrile: water]. The mixture was allowed to stand for 1 hr with intermittent sonication to ensure complete solubility of the drug, and then filtered through a 0.45 µm membrane filter, followed by adding diluent to obtain a stock solution of 2.5 mg/ml. Transfer 5ml of this solution to a 50 ml volumetric flask and made up to sufficient volume with dilutent to give an concentration of 250 µg/ml.

Linearity: Aliquots of standard Vildagliptin stock solution were taken in different 10 ml volumetric flasks and diluted up to the mark with the diluent such that the final concentrations of Vildagliptin are in the range of 50-300 mcg/ml. Each of these drug solutions (20 µL) was injected three time into the column, and the peak area and retention time were recorded. Evaluation was performed with PDA detector at 205 nm and a Calibration graph was obtained by plotting peak area versus concentration of Vildagliptin (Fig 2). The plot of peak area of each sample against respective concentration of Vildagliptin was found to be linear in the range of 50–300 µg/ml with correlation coefficient of 0.9999. Linear regression least square fit data obtained from the measurements are given in table I. The respective linear regression equation being Y= 33227.348x+101075.26. The regression characteristics, such as slope, intercept, and %RSD were calculated for this method and given in **Table I. Assay:** 20 µl of sample solution was injected into the injector of liquid chromatograph. The retention time was found to be 3.857 mins. The amount of drug present per tablet was calculated by comparing the peak area of the sample solution with that of the standard solution. The data are presented in **Table II**.

Recovery Studies: Accuracy was determined by recovery studies of Vildagliptin, known amount of standard was added to the pre-analysed sample and subjected to the proposed HPLC analysis. Results of recovery study are shown in Table II. The study was done at three different concentration levels.

RESULTS AND DISCUSSION

The system suitability tests were carried out on freshly prepared standard stock solution of Vildagliptin. Parameters that were studied to evaluate the suitability of the system are given in **Table III.**

Limit of Detection (LOD) and Limit of Quantification (LOQ): The limit of detection (LOD) and limit of quantification (LOQ) for Vildagliptin were found to be 0.25 and 0.75 µg/ml respectively. The signal to noise ratio is 3 for LOD and 10 for LOQ. From the typical chromatogram of Vildagliptin as shown in fig 1, it was found that the retention time was 3.857 min. A mixture of Acetonitrile and 0.03 M potassium dihydrogen phosphate in water (pH 3.5 adjusted with o-phosphoric acid) in the ratio of 80:20 v/v was found to be most suitable to obtain a peak well defined and free from tailing. In the present developed HPLC method, the standard and sample preparation required less time and no tedious extraction were involved.

A good linear relationship (r=0.9999) was observed between the concentration range of 50-300 mcg/ml. Low values of standard deviation are indicative of the high precision of the method. The assay of Vildagliptin tablets was found to be 98.56%. From the recovery studies it was found that about 101.10 % of Vildagliptin was recovered which indicates high accuracy of the method.

The absence of additional peaks in the chromatogram indicates non-interference of the common excipients used in the Tablets. This demonstrates that the developed HPLC method is simple, linear, accurate, sensitive and reproducible. Thus, the developed method can be easily used for the routine quality control of bulk and Tablets dosage form of Vildagliptin within a short analysis time.

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Table I: Linear Regression Data for Calibration curves.

Drug	Vildagliptin
Concentration range (mcg/ml)	50-300
Slope (m)	33227.348
Intercept (b)	101075.266
Standard Error of Estimate	57001.2
Correlation coefficient	0.9999
% RSD	0.42

Table II: Results of HPLC assay and Recovery studies

Sample	Amount claim Per tablet [mg]	% found by the Proposed method	% recovery*
1.	50	98.75	101.25
2.	50	98.56	101.50
3.	50	98.85	101.01

*Average of three different concentration levels.

Table III: Validation Summary

Validation Parameter	Results
System Suitability	
Theoretical Plates (N)	9733.95
Tailing factor	1.54
Retention time in minutes.	3.857
LOD (mcg/ml)	0.25
LOQ (mcg/ml)	0.75

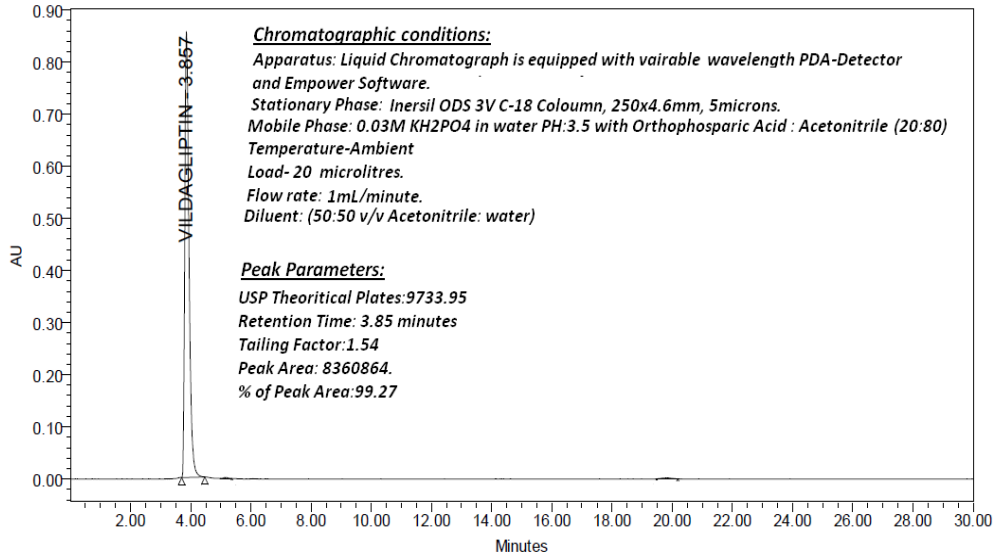

Fig 1: Typical Chromatogram of Vildagliptin by RP-HPLC.

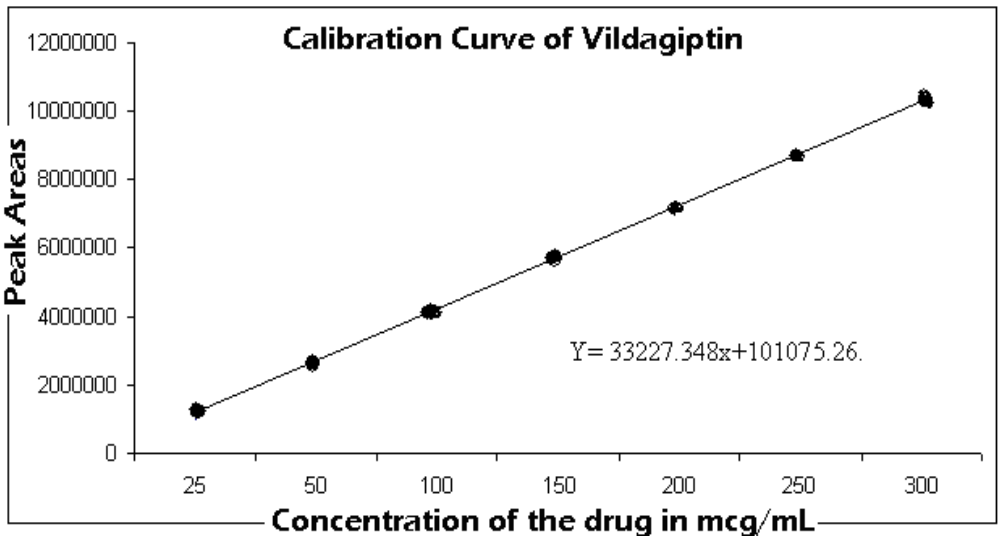

Fig 2: Calibration curve of Vildagliptin by RP-HPLC.

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