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Research Article

VALIDATED SPECTROSCOPIC METHOD FOR ESTIMATION OF SAXAGLIPTIN IN PURE AND FROM TABLET FORMULATION

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ABSTRACT

Simple, sensitive and cost effective UV-spectrophotometric method was developed for the estimation of saxagliptin in bulk and pharmaceutical formulations. Saxagliptin was estimated at 208 nm in methanol. Linearity range was found to be 5–40 µg/ml. Y= mx+c, r2 = 0.999. The apparent molar absorptivity was found to be 7.801×10^3 L mol^{-1} cm^{-1}. In the proposed method Sandell's sensitivity was found to be about 2.516×10^{-5}µg cm^{-2}/0.001. The LOD and LOQ were found to be 0.0607 µg/ml and 0.1821 µg/ml respectively. The developed method was validated respect to linearity, precision, accuracy.

Keywords: UV Spectrophotometry, Saxagliptin, Tablet analysis.

INTRODUCTION

Saxagliptin is chemically (1S,3S,5S)-2-[(2S)-2-amino-2-(3-hydroxy-1-adamantyl) acetyl]-2-azabicyclo[3.1.0]hexane-3-carbonitrile, previously identified as BMS-477118, is a new oral hypoglycemic (anti-diabetic drug) of the new dipeptidyl peptidase-4 (DPP-4) inhibitor class of drugs[1]. Saxagliptin is recently approved for the treatment of type 2 diabetes mellitus[2]. Literature survey reveals that the drug can be estimated only by LC-MS/MS[3] and no spectrophotometric methods have been reported. The present study describes a simple, sensitive, accurate and precise spectrophotometric method for estimation of saxagliptin in bulk and tablet formulation.

MATERIAL AND METHODS

Instrument used

The spectrophotometric measurements were carried out using An Elico UV/Visible double beam spectrophotometer SL - 164 with 1 cm matched quartz cells.

Reagents

Methanol of analytical grade was used. A pharmaceutical preparation of saxagliptin was obtained from local pharmacy.

Standard solution of the drug

A stock standard solution of 1000 µg mL^{-1} was prepared by dissolving saxagliptin in methanol. Working standard solution was then prepared by suitable dilution of the standard stock solution with methanol.

Determination of λmax

The working standard solution was subjected to scanning between 200 to 400 nm and absorption maximum was determined (Fig. 1). The λ max of saxagliptin was found as 208 nm and that was selected for the analysis. The calibration curve was prepared in the concentration range of 5-40 µg mL^{-1} at 208 nm. By using the calibration curve, the concentration of the sample solution can be determined.

Linearity and calibration

The aliquots working standard solution was diluted serially with sufficient methanol to obtain the concentration range of 5 – 40 µg mL^{-1}. A calibration curve for saxagliptin was obtained by measuring the absorbance at the λmax of 208 nm. Statistical parameters like the slope, intercept, coefficient of correlation, standard deviation and relative standard deviation were determined.

Procedure for formulations

Twenty tablets containing saxagliptin were weighed and finely powdered. An accurately weighed portion of the powder equivalent to 10 mg of saxagliptin was dissolved in a 50 ml of methanol and shaken well for about 5 minutes and filtered through a Whatman filter paper No.40. Convenient aliquots from this solution were taken for the determination of saxagliptin in the range 5 to 40 µg mL^{-1}.

Recovery studies

Recovery studies were performed to judge the accuracy of the method. Recovery studies were carried out by adding a known quantity of pure drug to a pre-analyzed formulations and the proposed method was followed. From the amount of drug found, percentage recovery was calculated. The results of analysis and recovery studies are given in Table 3.

RESULTS AND DISCUSSION

The UV scan of standard solution between 200 to 400 nm showed the absorption maxima at 208 nm, shown in fig. 1. The optical characteristics such as Beer's law limits, Sandell's sensitivity, molar absorptivity and the results are summarized in Table 1. The assay and precision studies results for tablets containing saxagliptin are shown in Table 2.The recovery studies for the formulation were studied and are shown in Table 3. The excellent recovery studies prove the accuracy of the method.

Table 1: Optical characteristics of proposed method

Parameters	Values
λ_{max} (nm)	208
Beer's law limit (µg mL^{-1})	5-40
Sandell's sensitivity (µg cm^{-2}/0.001 absorbance unit)	2.516×10^{-5}
Molar absorptivity (L mol^{-1} cm^{-1})	7.801×10^3
Regression equation (Y = mx + c)	
Slope (m)	0.027
Intercept(c)	-0.0189
Correlation coefficient (r^2)	0.999

Table 2: Assay results and precision studies

Formulation	Labeled amount (mg/ tablet)	(%) label claim* ± S.D	Precision** Repeatability	Inter-day	Intra-day
Saxagliptin Tablets	5	99.86 ± 0.3301	0.0028	0.0038	0.0021

* Average of six determinations. **SD of six determinations.

Table 3: Recovery study

Formulation	Label Claim (mg/ tablet)	(%) label claim* ± S.D	Amount of drug Added (mcg)	Amount of drug Recovered (mcg)	Percentage recovery* ± SD
Saxagliptin Tablets	5	99.86 ± 0.3301	12.0	11.99	99.97±0.2395
			15.0	14.99	99.97±0.2482
			18.0	18.03	100.18±0.2425

*Mean of six determinations.

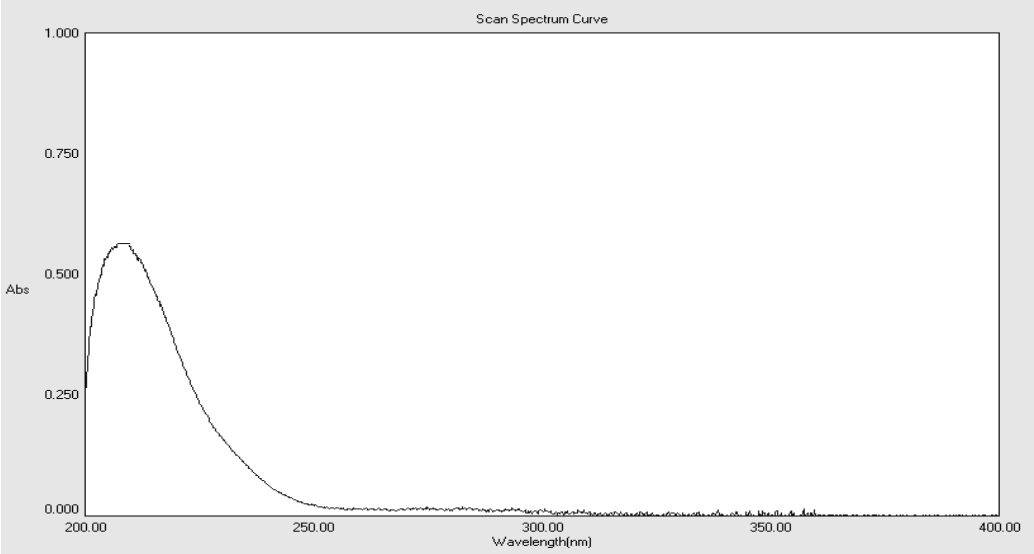

Fig. 1: UV spectrum of Saxagliptin in methanol

CONCLUSION

The proposed method was successfully applied for the determination of saxagliptin in pharmaceutical formulations. The results demonstrated that the procedure is accurate, precise and reproducible. This method can be applied for the estimation of saxagliptin in its dosage forms.

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