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Original Article

UV-SPECTROPHOTOMETRIC METHOD DEVELOPMENT AND VALIDATION FOR ESTIMATION OF SAXAGLIPTIN IN API AND PHARMACEUTICAL DOSAGE FORM

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ABSTRACT

Objective: A new sensitive, economical, simple, rapid UV spectrophotometric method has been developed for the estimation of Saxagliptin in API and pharmaceutical formulation.

Methods: Calibration curve method was used for the estimation of Saxagliptin in API and pharmaceutical formulation.

Results: The method was validated for linearity, range, accuracy, precision, robustness, LOD, LOQ. Linearity was found in the range of 10-60µg/ml. Accuracy was performed by using a recovery study. The amount of drug recovered was found to be in the range of 99.01-100.1%. All the parameters were validated as per the ICH guidelines.

Conclusion: This method is suitable forused routine analysis of present Saxagliptin in API and Pharmaceutical dosage form.

Keywords: Saxagliptin, UV spectrophotometer, Method validation, Methanol

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INTRODUCTION

Saxagliptin chemically is (1S, 3S, 5S,)-2-[(2S)-2-amino-2-(3-hydroxy-1-adamantyl)acetyl]-2-azabicyclo[3.1.0.] hexane-3-carbonitrile [1]. Molecular formula is $C_{18}H_{25}N_3O_2$, H_2O and the molecular weight is 333.43 [2]. Saxagliptin is an anti-diabetic drug and dipeptidyl peptidase-4 inhibitor class of the drug. Saxagliptin is breakdown of incretin hormones and increasing the level of these hormones on the body. Increase in incretin hormones production it gives beneficial action due to saxagliptin [2, 3]. Increasing insulin production in response to meals and gives the result of decreasing the amount of glucose that is liver produces. Saxagliptin is available in the form of tablets. Saxagliptin it is a white powder and freely soluble in methanol, acetonitrile, acetone, polyethylene glycol, ethanol [4, 5]. Literature survey carried out in that Saxagliptin has been estimated by HPLC, UV, LCMS/MS and stability method bye LC-MS [6, 7]. The present research study is a simple, sensitive, accurate and precise UV spectrophotometric method for the estimation of Saxagliptin in the API and its dosage forms with methanol as a solvent [8, 9].

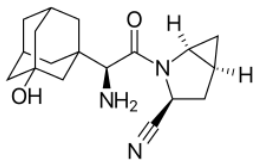

Fig. 1: Chemical structure of saxagliptin [2]

MATERIALS AND METHODS

Instrument and chemicals

A systronic UV-visible double beam spectrophotometer-2201 was used for the recording of spectra and measuring absorbance. Electronic analytical weighing balance (Shimadzu, AY220) is used for the weighing purpose. Sonicator (Oscar Ultrasonic Cleaner Microclean) it is used for the dissolving the saxagliptin into the methanol. Saxagliptin pure drug was gifted by Torrent Pharmaceutical Ltd. Ahmadabad, Gujarat. Tablets of 25 mg strength were purchased from the local pharmacy in Solapur and its brand name is Riax (Dr. Reddy's). Methanol is used as a solvent in this study.

Experimental work

Method development

Preparation of standard stock solution

Standard stock solution of Saxagliptin was prepared by transferring and accurately weight 10 mg Saxagliptin into 10 ml calibrated volumetric flask containing 6 ml of methanol and volume was made up to the mark by using a solvent to gives the concentration of 1000 µg/ml. Shaking well and solicitation was carried out for the uniform mixing. Form this take 2.5 ml of the solution was again transferred into 25 ml volumetric flask and make up the volume with methanol to give a concentration of 100 µg/ml it is a standard stock solution and further dilution was carried out with methanol to get concentration range of 10-60 µg/ml

Determination of absorption maxima

The standard stock solution of 100µg/ml was scanned in the range of 200-400 nm to determine the wavelength of maximum absorption. The drug showed maximum absorption at 213 nm.

RESULTS AND DISCUSSION

Method development [3]

Analytical method development it plays important role in pharmaceutical dosage forms. This graph indicates the identification of saxagliptin. Standard solution i.e. (50 µg/ml) of saxagliptin was scanned at 200-400 nm range in UV Visible spectrophotometer. Maximum absorbance was found to be at 213 nm.

The methods were validated for several parameters like Linearity, Accuracy, Precision, Robustness, Limit of Detection, Limit of Quantification and Specificity of saxagliptin.

Linearity and range

1, 2, 3, 4, 5, 6 ml of standard Saxagliptin solution was transferred into a series of 10 ml volumetric flask. The volume was made up to the mark with methanol to obtain the concentration of 10, 20, 30, 40, 50,

60µg/ml. The absorption of this solution was recorded and the graph of absorption against concentration was plotted. The correlation coefficient (r^2) of the least squares linear regression of Saxagliptin was calculated. Range is 10-60. (µg/ml) was taken (fig. 3)

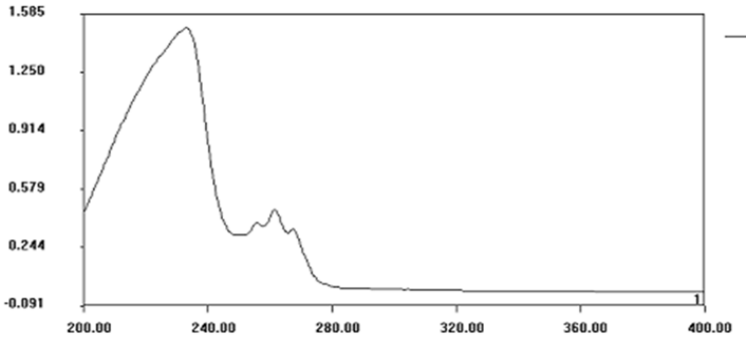

Fig. 2: UV visible spectrophotometer graph

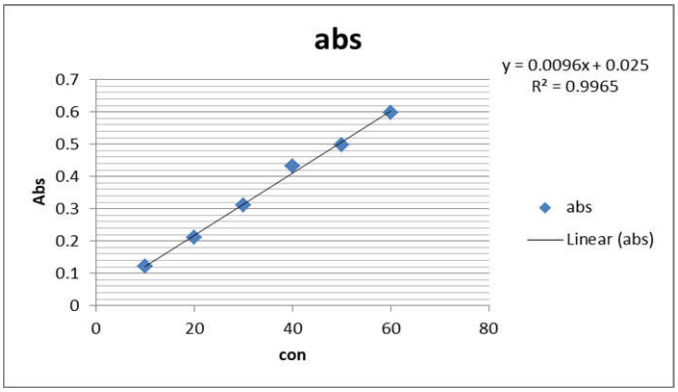

Fig. 3: Calibration curve for saxagliptin

The wavelength for linearity was scanned at 213 nm. By using five different concentrations for linearity the regression coefficient was found to be 0.996 i.e. in the limit of standard. Hence linearity was found to be validated.

Accuracy

The recovery study was carried out by the standard addition method by adding the known amount of Saxagliptin to the pre-analyzed sample at three different concentration level that are 80%, 100%, 120% of assay concentration and percent recovery were calculate (table 1)

% Recovery =observed value/true value*100

Precision

The precision of the method was determined in term of repeatability, intra-day precision and inter-day precision (Intermediate precision). Intra-day precision was determined by analyzing the drug at concentration (40µg/ml) and each concentration for these two times on the same day. Inter-day precision was determined similarly, but the analysis is carried out daily for two consecutive days.

Repeatability: Repeatability of the method was determined by analyzing six samples of the same concentration of the drug 40µg/ml. The absorbance of each was measured (table 2 and 3)

Table 1: Result of accuracy

Name of drug	Recovery levels	Concentration (µg/ml)	Amount recovered	% Recovery with SD
Saxagliptin	80%	30	30.002	100.02±0.80
	100%	40	40.001	100.01±0.26
	120%	50	50.004	100.02±0.5

Table 2: Results for Intra-day morning precision

S. No.	Concentration (µg/ml)	Absorbance	SD	%RSD
1.	40	0.431		
2.	40	0.430	0.001049	0.243061
3.	40	0.432		
4.	40	0.431		
5.	40	0.433		
6.	40	0.432		

Table 3: Results for Inter-day precision

S. No.	Concentration (µg/ml)	Absorbance	SD	%RSD
1.	40	0.432		
2.	40	0.431	0.000753	0.17432
3.	40	0.434		
4.	40	0.432		
5.	40	0.433		
6.	40	0.431		

For Intraday and inter-day precision, relative standard deviation is in limit i.e. less than 2%; hence parameter is validated.

Robustness

The robustness of the developed method is its capacity to remain unaffected by small changes in altered concentration. To determine the robustness of the method, the wavelength was studied at±5 nm (table 4)

Ruggedness

Ruggedness was determined by carrying out the analysis by two different analysts and the respective absorbance was noted and the results were indicated as % RSD.

Table 4: Results for robustness

S. No.	Wavelength	Absorbance	SD	%RSD
1.	213	0.121		
		0.123	0.001	0.81967
		0.122		
2.	215	0.123		
		0.124	0.0020	1.7016
		0.120		

By change of concentration and wavelength i.e. 213 nm and 215 nm % RSD is less than 2% i.e. within the range. So the parameter was validated.

Table 5: Results for ruggedness

Concentration	Analyst 1	Analyst 2
40(µg/ml)	0.432	0.431
	0.431	0.431
	0.431	0432
	0.432	0.433
	0.431	0.432
	0.433	0.431

By change in analyst and laboratory, there is no effect on absorbance with the same conditions (table 5)

Limit of detection

Limit of detection is the lowest amount of the sample that can be detected. It is not that much necessarily to quantitate as a true amount. The LOD was found to be 0.3967(µg/ml).

LOD=3.3(SD/S)

Where, SD-standard deviation; S-slope of the curve

Limit of quantitatication

Limit of Quantitatication of an individual analytical procedure is the lowest amount of analyte in the sample, which can be quantitated as an exact value. The LOQ was found to be 1.202(µg/ml).

LOQ=10(SD/S) Where, SD-standard deviation; S–slope of the deviation.

CONCLUSION

The proposed UV spectroscopic method was found to be accurate, precise, stable linear, specific, and simple for quantitative estimation of saxagliptin in bulk and pharmaceutical dosage form. Hence the present UV spectroscopic method is suitable for routine analysis of saxagliptin and pharmaceutical formulation.

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AUTHORS CONTRIBUTIONS

All the authors have contributed equally.

CONFLICT OF INTERESTS

Declared none

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