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VALIDATED SPECTROPHOTOMETRIC METHOD FOR THE QUANTITATION OF SITAGLIPTIN IN BULK AND TABLET DOSAGE FORM

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ABSTRAC

Simple, precise and accurate area under curve spectroscopic method has been developed and validated for the estimation of Sitagliptin in bulk and Pharmaceutical dosage form. The drug shows maximum absorption (λ_{max}) at 267nm in 0.1N Sulphuric acid solution and Area under Curve [AUC] in absorption spectra was measured between the wavelength range 262 to 272nm which obeys Beer's law in the concentration range of 10-50µg/ml. The linearity study was carried out and regression coefficient was found to be 0.9999 and it has showed good linearity, precision during this concentration range. The % recovery was found to be 98.85-99.1. The LOD and LOQ was found to be 0.4472 and 1.35µg/ml. The % relative standard deviation was found to be less than 2. As per ICH guidelines the method has been validated for linearity, precision, accuracy, robustness, ruggedness, LOD and LOQ. The developed and validated method can be successfully applied for reliable quantification of Sitagliptin in bulk and pharmaceutical dosage form.

KEYWORDS: - Sitagliptin, Area under curve spectroscopy, Validation, Pharmaceutical formulations.

INTRODUCTION

Sitagliptin is a anti-diabetic medication used to treat type 2 diabetes. Sitagliptin is a dipeptidyl peptidase-4 inhibitor which is used in the combination with diet and exercise, either alone or in the combination with other oral hypoglycemic agents.[1]

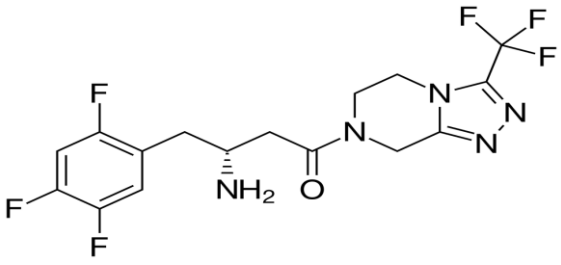

Fig. 1: Chemical structure of sitagliptin.

Literature survey revealed that there were few analytical methods have been reported for the determination of Sitagliptin in pure drug and pharmaceutical dosage forms by using UV spectrophotometric,[2-8] HPLC,[9-20] and HPTLC,[21] so far.

The aim of present work is to develop and validate a novel, rapid, simple, precise and specific Area under curve Spectrophotometric method for estimation of Sitagliptin in bulk and tablet dosage form.

MATERIALS AND METHODS

Instrument: Ultra Violet-Visible double beam spectrophotometer, SHIMADZU (model UV-1800) with UV probe software. All weights was taken on analytical balance.

Chemicals: Sitagliptin pure drug was obtained as a gift sample from Recipharma Ltd. Nelamangala, Bangalore. and its pharmaceutical dosage form Sitagliptin 20 tablet labelled claim 100mg from local pharmacy manufactured by Recipharma Ltd.

Solvent: 0.1N Sulphuric Acid (prepared by dissolving 2.72ml in 1000ml of distilled water).

Selection of analytical wavelength: Appropriate dilutions of Sitagliptin was prepared from standard stock solution and using spectrophotometer solution was scanned in the wavelength range 200-400nm. Area under Curve [AUC] in absorption spectra was measured between the wavelength range 262-272nm as the wavelength for detection (Fig-2).

Preparation of standard stock solution: 100mg of Sitagliptin was weighed accurately and transferred in to 100ml volumetric flask and diluted in 0.1N Sulphuric acid up to mark. From this, the solution was further diluted into 100µg/ml and pipette out 1, 2, 3, 4, and 5ml into 10ml individual volumetric flask and dilute in 0.1N Sulphuric acid up to mark, this gives 10, 20, 30, 40, and 50µg/ml concentration.

Preparation of sample solution: 20 tablets of Sitagliptin marketed formulations was weighed and powdered. A quantity of tablet powder equivalent to 100mg of Sitagliptin was transferred into a 100ml of volumetric flask then it was diluted with 0.1N Sulphuric acid and made up to the mark.

METHOD AND VALIDATION

The method was validated according to ICH guidelines.

RESULTS AND DISCUSSION

Method: Area under curve spectroscopy**.**

Linearity: The linearity of an analytical method and its capacity to show the test results that are directly proportional to the concentration of the analyte in the sample within the range. The linearity was established in the range of 10-50µg/ml and Area under Curve[AUC] in absorption spectra was measured between the wavelength of 262 to 272nm as absorbance values are shown in table-1 (Fig-3). The calibration curve was prepared by plotting graph v/s concentration and absorbance and therefore the graph shown in (Fig-4). Statistical parameter like slope, intercept, regression equation, correlation coefficient and Sandell's sensitivity were determined. (table-2).

Precision: The precision of an analytical method express the closeness of a series of individual analyte measurements obtained from multiple sampling of the equivalent sample. This method was done by intra-day and inter-day studies. Intra-day precision was determined by analysing the same concentration for six times in a same day. Inter-day precision was determined by analysing the same concentration daily for six days. (table-3).

Accuracy: The accuracy of an analytical method show that closeness of test results obtained by that method to the true value. To assess the accuracy of the developed method, recovery studies was carried out at three different levels as 80%, 100% and 120%. In which the formulation concentration kept constant and different pure drug concentration. (table-4).

Ruggedness: The ruggedness is the reproducibility of results when the method is performed under the different conditions. This involves different analyst, laboratories, instruments, temperature etc. Ruggedness was determined between distinct analyst, the value of %RSD was found to be less than 2. (table-5).

LOD and LOQ: The limit of detection is an discrete analytical method is the smallest amount of analyte in a sample which can be reliably detected by the analytical method. The limit of quantitation is an discrete analytical procedure of the smallest amount of analyte in a sample which can be quantitatively determined. LOD and LOQ was calculated by using following formula.

LOD = 3.3(SD)/S and LOQ = 3(LOD)

LOD and LOQ value of Sitagliptin was found to be 0.447 and 1.35µg/ml.

TABLES

Table 1: Results of calibration curve at 262-272nm by area under curve method.

Sl. no.	Concentration in µg/ml	Absorbance ± Standard deviation*
1	0	0
2	10	0.047±0.00137
3	20	0.095±0.00211
4	30	0.142±0.00115
5	40	0.189±0.00058
6	50	0.234±0.00094

**Average of six determinations.*

Table 2: Regression parameter for sitagliptin at 262-272nm by area under curve method.

Regression parameter	Results
Range(µg/ml)	10-50
λ_{max}(nm)	262-272
Regression Equation	Y= 0.0047x+0.0005
Slope(b)	0.0047
Intercept(a)	0.0005
Correlation coefficient(r^2)	0.9999

Sandell's equation	0.211
Limit of detection(µg/ml)	0.447
Limit of quantitation(µg/ml)	1.35

Table 3: Determination of precision results for sitagliptin at 262-272nm by area under curve method.

Concentration (µg/ml)	Intra-day Absorbance ±Standard deviation*	%RSD**	Inter-day Absorbance ±Standard deviation*	%RSD**
10	0.048±0.00089	1.8	0.047±0.00057	1.22
20	0.097±0.00068	0.70	0.096±0.000687	0.71
30	0.141±0.00076	1.5	0.142±0.000745	0.52
40	0.186±0.00146	0.78	0.189±0.000957	0.50
50	0.233±0.001344	0.57	0.233±0.000816	0.35

*****Average of six determinations, **percentage relative standard deviation*

Table 4: Determination of Accuracy results for Sitagliptin at 262-272nm by Area under curve method.

Spiked Levels	Amount of Sample (µg/ml)	Amount of Standard (µg/ml)	Amount Recovered	% Recovery ±Standard deviation*	%RSD**
80	30	24	53.4	98.85%±0.397	0.401
100	30	30	59.4	99.1%±0.734	0.741
120	30	36	65.7	99.68%±0.014	0.014

*****Average of six determinations, **percentage relative standard deviation.*

Table 5: Determination of ruggedness results for sitagliptin at 262-272nm by area under curve method.

Analysts	Analyst 1	Analyst 2
Mean absorbance	0.189	0.190
±Standard deviation*	0.000943	0.0011
%RSD	0.049	0.601

**Average of six determinations, **percentage relative standard deviation.*

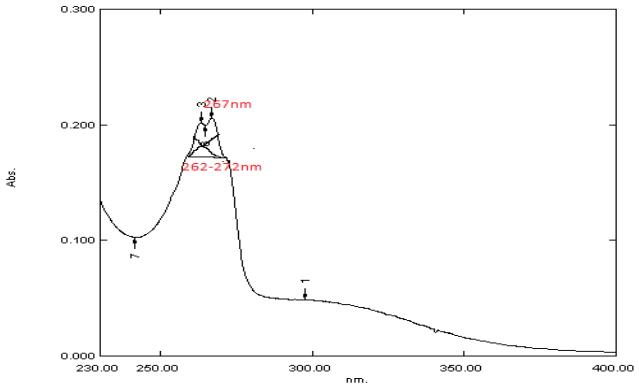

Fig. 2: Area under curve spectrum of sitagliptin at 262-272nm.

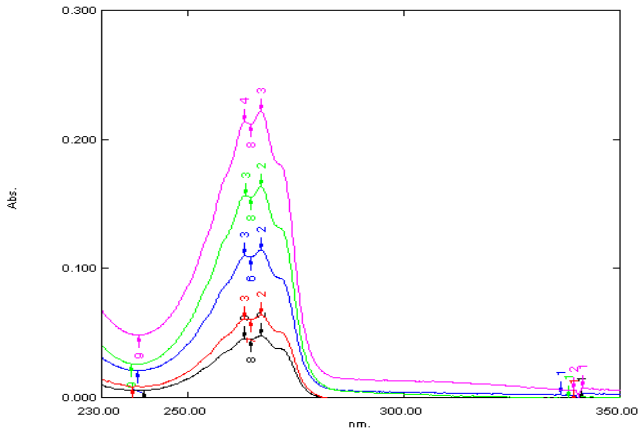

Fig. 3: Area under curve overlain spectra of sitagliptin showing absorbance at 262-272nm.

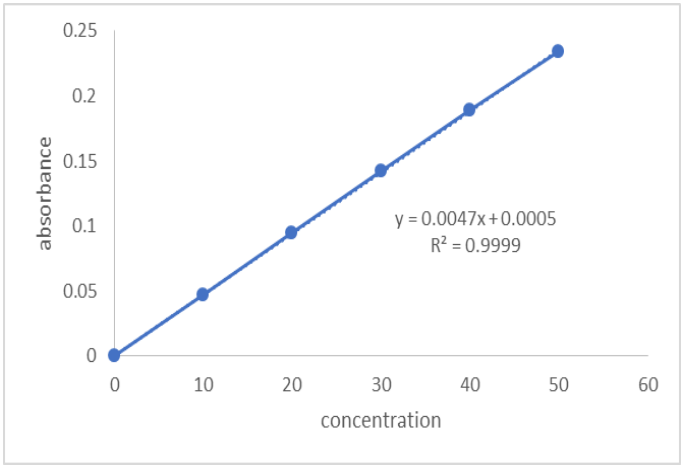

Fig. 4: Calibration curve of sitagliptin at 262-272nm by area under curve method.

CONCLUSION

As per ICH guidelines, the developed analytical method was carried meets the acceptance criteria. It was concluded that method is simple, specific, accurate, economical and sensitive and can be used for routine analysis of Sitagliptin in bulk drug and in pharmaceutical dosage forms.

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