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

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ABSTRACT

Simple, precise and accurate zero order derivative 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 obeys Beer's law in the concentration range of 10-50µg/ml. The linearity studywas 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 98.49-99.98. The LOD and LOQ were found to be 0.251 and 0.761µg/ml. The % relative standard deviation wasfoundto 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 bulkand pharmaceutical dosage form.

KEYWORDS

Sitagliptin, Zero order derivative spectroscopy, Validation and Pharmaceutical formulations.

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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].

Literature survey revealed that there was few analytical methods have been reported for the determination of Sitagliptin in pure drugand 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 Zero order derivative UV Spectrophotometric method for estimation of Sitagliptin in bulk and tablet dosage form.

MATERIAL 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 fromRecipharma 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. The absorption spectraobtained and shows maximum absorbance at 267nm. And it was selected as the wavelength for detection (Figure No.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 solution was further diluted into 100µg/ml and from this solution 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 were 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 Acidand made up to the mark.

Method and validation

The method was validated according to ICH guidelines.

RESULTS AND DISCUSSION

Method

Zero order derivative 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 was measured at 267nm and absorbance values are shown in Table No.1. The calibration curve was prepared by plotting graph of concentration verses absorbance and the graph shown in Figure No.3. Statistical parameter like slope, intercept, regression equation, correlation coefficient and sandell's sensitivity were determined. (Table No.2).

Precision

The precision of an analytical method express the closeness of a series of individual analyte measurements obtained from multiple sampling of the same sample. Precision was established by intra-day and inter-day study. Intra-day precision was determined by analysing the same concentration for three times in a same day. Inter-day precision was established by analysing the same concentration daily for three days. (Table No.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 were carried out at three different level as 80%, 100% and 120%. In which the formulation concentration kept constant and different pure drug concentration. (Table No.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 No.5).

Limit of detection and Limit of Quantitation

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 established. LOD and LOQ was calculated using following formula.

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

LOD and LOQ value of Sitagliptin was found to be 0.251 and 0.761µg/ml.

Table No.1: Results of calibration curve at 267nm by zero order spectroscopy

S.No	Concentration in µg/ml	Absorbance ±Standard deviation*
1	0	0
2	10	0.046±0.00149
3	20	0.095±0.00179
4	30	0.142±0.00195
5	40	0.190±0.00221
6	50	0.235±0.0045

*Average of six determinations.

Table No.2: Regression parameter for Sitagliptin by zero order spectroscopy

S.No	Regression parameter	Results
1	Range (µg/ml)	10-50
2	λ_{max}(nm)	267
3	Regression Equation	Y= 0.0047x+0.001
4	Slope (b)	0.0047
5	Intercept (a)	0.001
6	Correlation coefficient (r^2)	0.9999
7	Sandell's equation	0.211
8	Limit of detection (µg/ml)	0.251
9	Limit of quantitation (µg/ml)	0.761

Table No.3: Determination of precision results for Sitagliptin at 267nm by zero order spectroscopy

S.No	Concentration (µg/ml)	Intra-day Absorbance ±Standard deviation*	%RSD**	Inter-day Absorbance ±Standard deviation*	%RSD**
1	10	0.045±0.0018	1.511	0.045±0.00074	1.55
2	20	0.094±0.0017	1.80	0.094±0.00157	1.59
3	30	0.143±0.00094	0.65	0.142±0.00203	1.4
4	40	0.191±0.00197	0.99	0.190±0.0022	1.57
5	50	0.238±0.0020	0.840	0.238±0.0035	1.470

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

Table No.4: Determination of Accuracy results for Sitagliptin at 267nm by Zero order spectroscopy

S.No	Spiked Levels	Amount of Sample (µg/ml)	Amount of Standard (µg/ml)	Amount Recovered	% Recovery ±Standard deviation*	%RSD**
1	80	30	24	53.21	98.49% ±0.410	0.416
2	100	30	30	59.1	98.8% ±0.424	0.425
3	120	30	36	65.92	99.8% ±0.080	0.080

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

Table No.5: Determination of Ruggedness results for Sitagliptin at 267nm by Zero order spectroscopy

S.No	Analysts	Analyst 1	Analyst 2
1	Mean absorbance	0.190	0.191
2	±Standard deviation*	0.000957	0.000957
3	%RSD	0.498	0.499

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

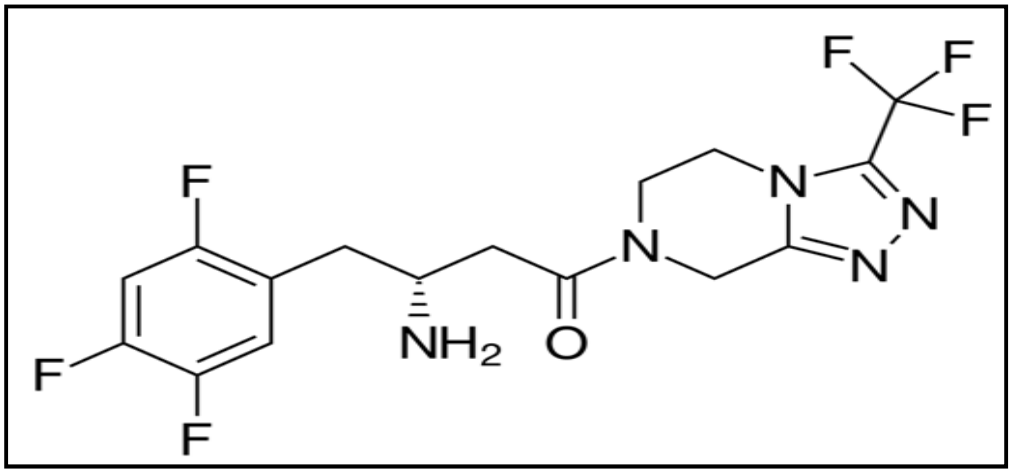

Figure No.1: Chemical structure of Sitagliptin

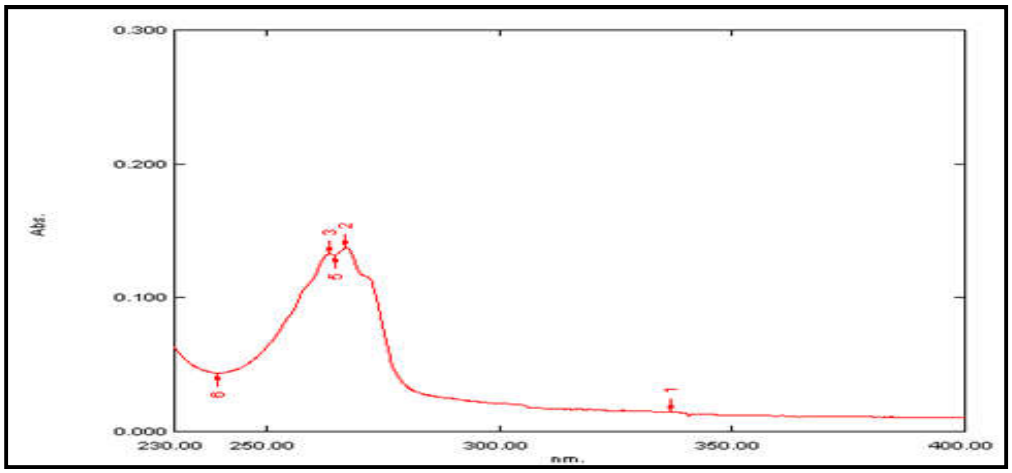

Figure No.2: Zero order spectrum of Sitagliptin at 267nm

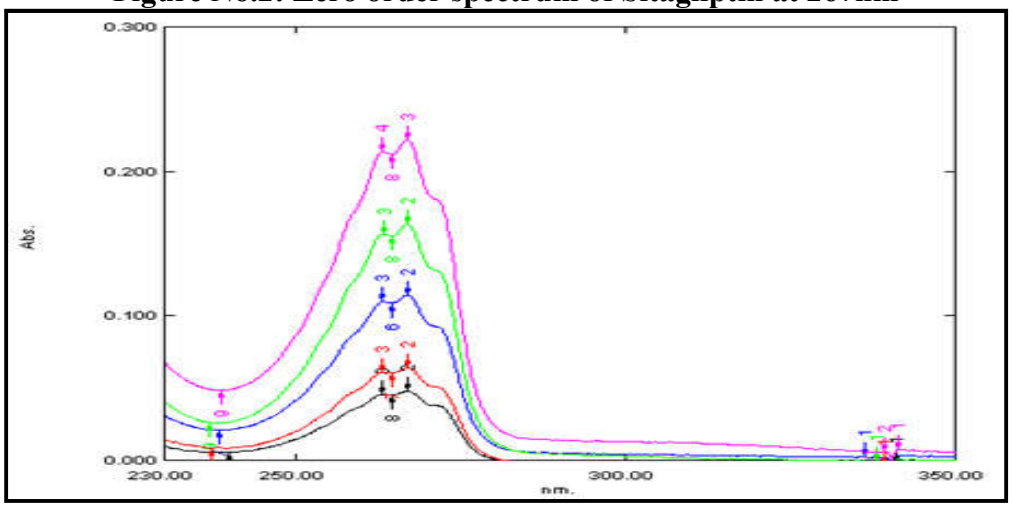

Figure No.3: Zero order overlain spectra of Sitagliptin showing absorbance at 267nm

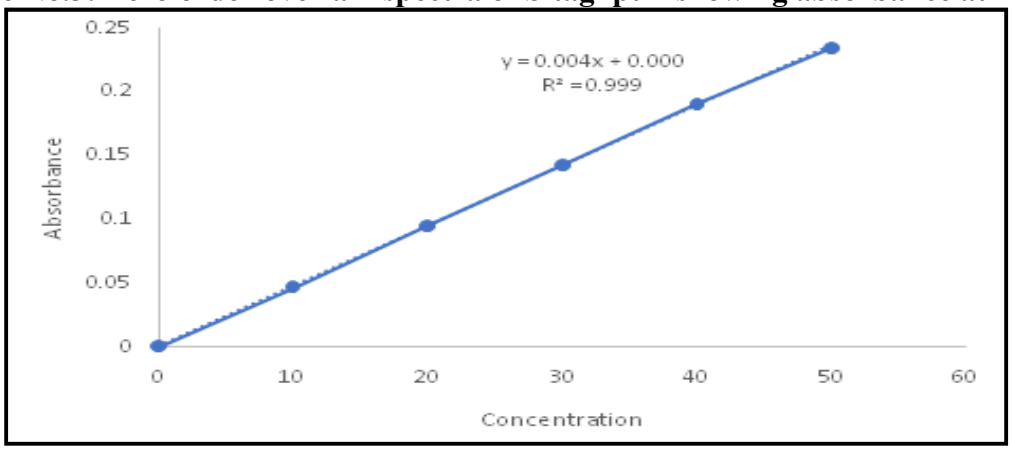

Figure No.4: Calibration curve of Sitagliptin by zero order spectroscopy

CONCLUSION

As per ICH guidelines, the developed analytical method meets the acceptance criteria. It was concluded that the 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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CONFLICT OF INTEREST

We declare that we have no conflict of interest.

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