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Development and validation of UV spectrophotometric method for estimation of sitagliptin phosphate

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ABSTRACT

Sitagliptin phosphate is antidiabetic drug. A sensitive, precise, accurate and simple UV spectrophotometric method has been developed for Estimation of Sitagliptin phosphate in tablet dosage form. The quantification was achieved by the spectroscopy method at 267nm for Sitagliptin phosphate. Sitagliptin phosphate(R^2=0.9934) shows Linearity in a concentration range of 5-40µg /ml LOD value was found to be 0.139µg/mL and LOQ value found to be 0.422µg/mL respectively. The results of analysis have been validated statistically and recovery studies carried out in the range 80-120% to confirm the accuracy of the proposed method. The relative standard deviation was found to be <2.0%. The Proposed method is recommended for routine analysis since it is rapid, simple, accurate and also sensitive and specific.

Key Words: Sitagliptin phosphate, spectrophotometry, Linearity, Validation.

INTRODUCTION

Sitagliptinphosphate (SITA) is chemically((R)-4-oxo-4-[3(trifluoromethyl)-5,6 dihydro[1,2,4]triazolo[4,3-a]pyrazin-7(8H)-yl]-1-(2,4,5-trifluorophenyl)butan-2-amine) is an oral antihyperglycemic (ant diabetic drug) of the dipeptidyl peptidase-4 (DPP-4) inhibitor class. Competitively inhibit the enzyme dipeptidyl peptidase 4 (DPP-4). Involved in the breakdown of incretins such as glucagonlikeparticle-1 (GLP-1) which potentiate insulin secretion in vivo. Inhibition of DPP-4 reduces the breakdown ofGLP-1 and increases insulin secretion; this suppresses the release of glucagon from the pancreas and drives down blood sugar levels. This drug is not official in any pharmacopoeia.

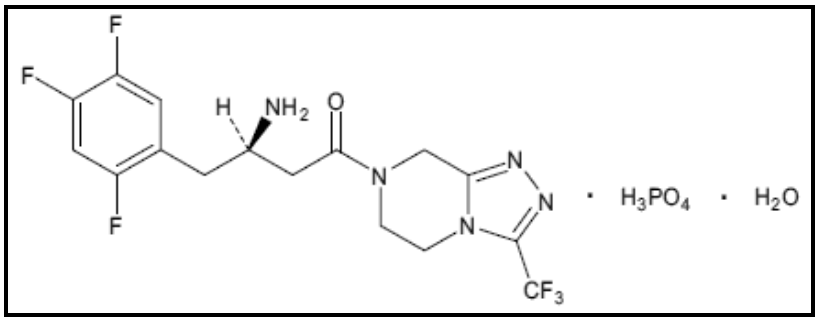

Fig.1: Chemical structure of sitagliptinphosphate (STA)

A comprehensive literature research reveals the lack of a spectrophotometric analytical method for Sitagliptin in pure drug and in tablet dosage form pharmaceutical formulations.

MATERIALS AND METHODS

MATERIALS:

A Jasco V-630 UV- Visible double beam spectrophotometer with 1 cm matched Quartz cells were used for spectral measurement. Schimadzu AX 200 Analytical balance was used for weighing purposes. The Reference Standard of Sitagliptin phosphate (SITA) was kindly provided by Watson pharmaceuticals and tablet (Januvia) was procured from market was utilized for the study. All chemicals and reagent used were of analytical grade.

Selection of common solvent:
Main criteria for media selection were solubility and stability. The media used in reported method was 0.1N Hcl. Hence 0.1N Hcl selected as analytical media for present work.

Preparation of 0.1N HCl :
8.5ml of 35% hydrochloric acid was accurately measured and transferred into a 1000ml volumetric flask and the volume was made up to the mark with distilled water.

Preparation of stock solution:
Standard stock solutions containing Sitagliptin phosphate (SITA) was prepared by dissolving 10 mg in 50 ml of 0.1 N HCl, It was then sonicated for 10 minutes and the final volume the solution was made up to 100 ml with same solvent to get stock solutions containing 100µg/ mL of SITA a 100 ml volumetric flasks.

Determination of absorption maxima:
A 50 µg/ml solution of SITA was prepared and scanned in UV range of 200-400nm and spectrum was obtained (Figure 2). The λ max was found to be at 267nm where the absorbance was found to be maximum.

Direct spectrophotometric Method:
The aliquots were prepared from stock solution of Sitagliptin phosphate by appropriate dilution in 0.1N Hcl to obtain the concentration 5-40 µg/ml. All the solutions were scanned from 400-200nm.

Application of proposed method to Tablet formulation
Twenty tablets of SITA (each tablet containing 25 mg of Sitagliptin phosphate) were weighed and finely powdered. Accurately weighed powder samples equivalent to 10 mg of SITA transferred to a 100 ml volumetric flask. About 50 ml of solvent 0.1 N HCl was added to the flask and placed in an ultrasonic bath at room temperature for 15 min. The solution was cooled and made up to volume and then filtered through 0.45 µmwhatman filter paper. Resulting solution was equivalent to 100 µg/ml SITA. Accurately measured 1ml of filtrate transferred to 10 ml volumetric flask and diluted up to the mark. The resultant solution was scanned. The absorbance's was recorded at 267nm. The content of SITA was calculated and % labeled claim was determined and results shown in **Table 1**

Validation:
The method was validated according to ICH Q2B guidelines for validation of analytical procedures in order to determine the linearity, sensitivity, precision and accuracy for the analyte.

Accuracy:
To ascertain the accuracy of the proposed methods, recovery studies were carried at three different levels (80%, 100% and 120%). Percent recovery for SITA by this method, was found in the range of

Linearity:
The linearity of measurement was evaluated by analyzing different concentration of the standard solution of Sitagliptin(5-40 µg/ml) and calibration curves are shown in (figure.-3)

Precision
To access the repeatability of the present study the Intra-day and inter-day precision of the method was evaluated for SITA at three different independent concentrations by determining their assay.[Table 3]

Limit of Detection (LOD) and Limit of Quantization (LOQ):
The LOD and LOQ of Sitagliptinby proposed methods were determined using calibration standards. LOD and LOQ were calculated as 3.3σ/S and 10σ/S respectively, where S is the slope of the calibration curve and σ is the standard deviation of response. The results of the same are shown in Table 3.

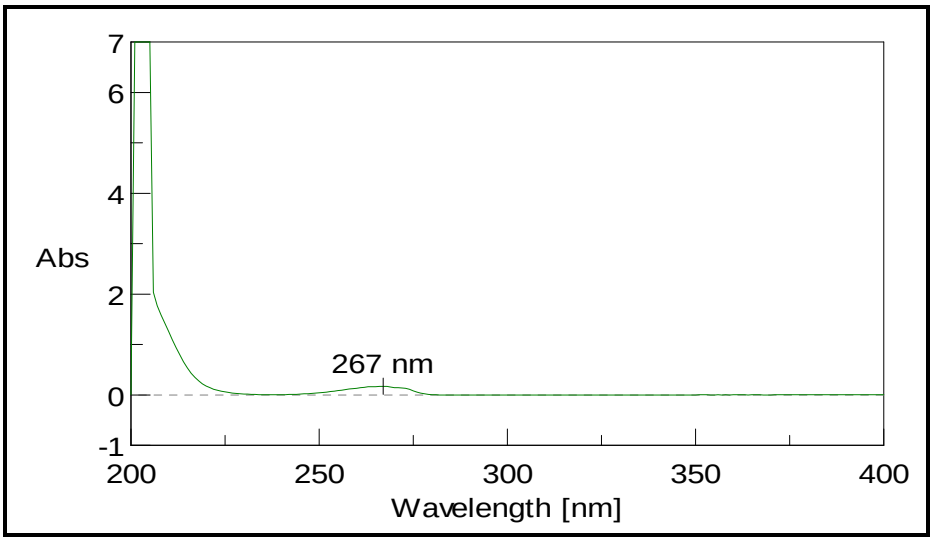

Fig. 2: UV spectra of GLM

Table 1: Analysis of Tablet formulation

Drug	Label claim (mg/tab)	% Drug found	±SD
SITA	25	99.58	1.04

(n=6)

Table 2: Recovery study of SITA

Drug	Level of addition (%)	Amount added (µg/ml)	Amount recovered (µg/ml)	%Recovery ± SD
SITA	80	8	8.15	100.18± 1.56
	100	10	9.97	99.09± 1.38
	120	12	11.88	99.87± 1.93

(n=3)

Table 3: Optical characteristics data and validation parameters

Parameters	Values for SITA
Absorption maxima (λ max)	267nm
Beer's law limit (µg/ml)	5-40
Regression equation	y = 0.0044x + 0.0031
Correlation coefficient (R2)	R^2 = 0.9934
Molar absorptivity	0.27×10^2
Accuracy (%Recovery ± SD)	100.14 ± 1.04
Precision	
Intraday*(%RSD)	99.65± 1.55
Interday*(%RSD)	99.88± 1.62
LOD (µg/ml)	0.139
LOQ (µg/ml)	0.422

**(n=6)*

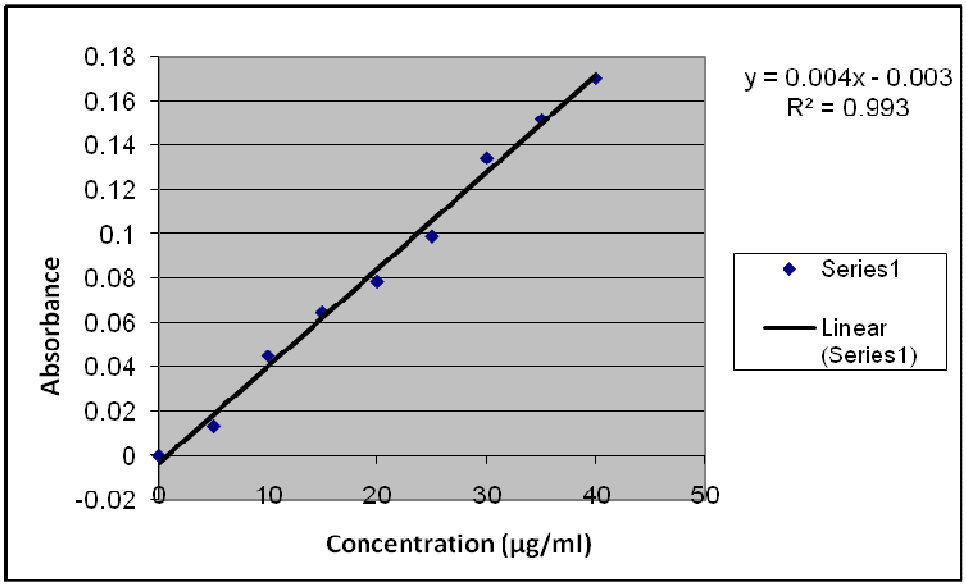

Fig. 3: Calibration curve SITA

RESULTS AND DISCUSSION

Under experimental conditions described, calibration curve, precision and recovery studies were performed. The drug obey beer's law in the concentration range 5-40 µg/ml the proposed method with good correlation coefficient. The results of tablet formulation analysis are presented in Table 1. Results of recovery studies are shown in Table 2. The accuracy and reproducibility is evident from the data as results are close to 100 % and low standard deviation. The proposed method is simple, economical, rapid, precise and accurate. Hence these can be used for routine analysis of SITA in bulk and tablet formulation.

CONCLUSION

The proposed method is simple and accurate for estimation of SITA. The most striking feature of method should be its simplicity, economy and rapidity, which is always preferred by an analyst. The described methods give accurate and precise results for determination of SITA hence can be used for routine analysis.

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