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Development and validation of UV- Spectroscopic method for estimation of Voglibose in bulk and tablets

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Abstract

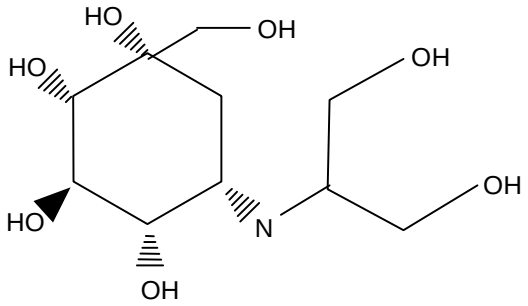
Simple, rapid, sensitive and accurate UV- Spectrophotometric methods have been developed for estimation of Voglibose in pharmaceutical formulation. Since Voglibose only absorbs UV in the low wavelength region, it cannot be detected with high sensitivity. So special detection methods are necessary for analysis of Voglibose.Voglibose shown effective results for various analytical instruments only in the combination of Taurine and Sodium periodate. Drug solution was derivatized using Taurine and Sodium periodate in water and methanol. Drug exhibited distinct λ_{max} in methanol at 282nm. Linearity was observed in the concentration range 10-80 µg/ml. (r^2 = 0.997). Amounts of drug estimated from tablet formulation were in good agreement with label claim. The method was validated statistically and by recovery studies. The proposed methods are economical and sensitive for the estimation of voglibose in bulk and tablet dosage forms.

Key words: Voglibose, UV- Spectrophotometry, oral antidiabetic agent, derivatization, estimation, validation.

Introduction

Voglibose 3,4-Dideoxy-4-[2-hydroxy-1-(hydroxyl methyl) ethyl]amino-2-c-(hydroxymethyl)-D-epiinositol, has attracted considerable interests due to its wide range of therapeutic and pharmacological properties, including its excellent inhibitory activity against α-glucosidase and its action against hyperglycemia and various disorders caused by hyperglycemia. Voglibose, a new potent glucosidase inhibitor used for type 2 diabetes, has shown strong anti-obesity and anti-diabetic activity. As a glucosidase inhibitor, the compound exerts its activity within the

gastrointestinal tract of humans. The drug delays glucose absorption and thus, reduces the post-prandial blood glucose peaks [1-3]. Voglibose obtained from organic synthesis processes is similar to structurally related carbohydrates found naturally [4, 5] and has the empirical formula $C_{10}H_{21}NO_{7.}$ Since most carbohydrates lack chromophore and/or flurophore groups, their analysis by liquid chromatography (LC) often requires derivatization procedures [6]. Since Voglibose only absorbs UV in the low wavelength region, it cannot be detected with high sensitivity. So special detection methods are necessary for analysis of voglibose. Voglibose shown effective results for various analytical instruments only in the combination of Taurine and Sodium periodate [7]. Drug solution was derivatized using Taurine and Sodium periodate in water and methanol. Drug exhibited distinct λ_{max} in methanol.



Chemical structure of Voglibose

Analysis is an important component in the formulation development of any drug molecule. A suitable and validated method has to be available for the analysis of drug(s) in the bulk, in drug delivery systems, from release dissolution studies and in biological samples. If a suitable method, for specific need, is not available then it becomes essential to develop a simple, sensitive, accurate, precise, reproducible method for the estimation of drug samples. No Spectrophotometric method for estimation of voglibose in bulk and formulations. Our main concern is development and validation of UV spectrophotometric method as per ICH guidelines. Thus the present study was undertaken to develop and validate a simple, sensitive, accurate, precise and reproducible UV method for voglibose [8].

Materials and Methods

Instrument used were JASCO V-630 double beam UV/Visible Spectrophotometer with matched pair of quartz cell (1.0cm path) was employed for absorption measurements [9-20]. Voglibose pure drug was obtained from Ranbaxy Research Laboratories as gift sample and was used without further purification. All chemicals and reagents used were of analytical grade. Voglibose tablets were purchased from market.

Preparation of standard stock solution

Standard drug solution of Voglibose was prepared by dissolving 10mg Voglibose in 100 ml of methanol to get a concentration of 100 µg/ml. The solution was derivatized with Taurine and Sodium periodate.

Selection of solvent

Absorbance of the drug was higher and drug exhibited distinct λ_{max} in methanol and hence methanol was selected as solvent for further studies.

Selection of wavelength

The 10 µg/ml of standard solution (derivatized) was scanned between 200-400 nm and found that the peak at 282 nm showed maximum absorption.

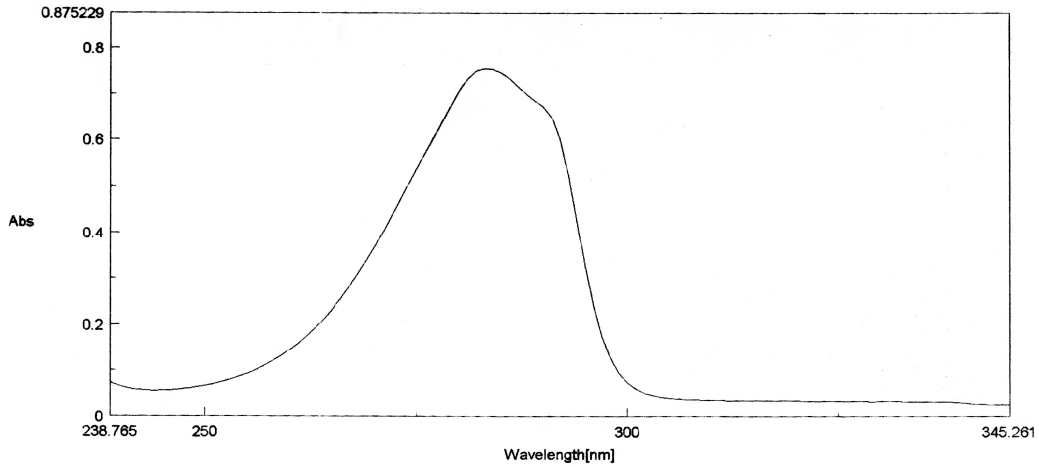


Figure 1: Selection of wavelength

Preperation of calibration curve

Calibration curve was prepared at λ_{max} 282 nm using JASCO UV- Visible Spectrophotometer. For this stock solution of 100 µg/ml was prepared. Serial dilution of Voglibose stock solution were prepared and scanned in the range range of 200-400 nm. Absorbance was taken at λ_{max} 282 nm against blank. The calibration curve was plotted.

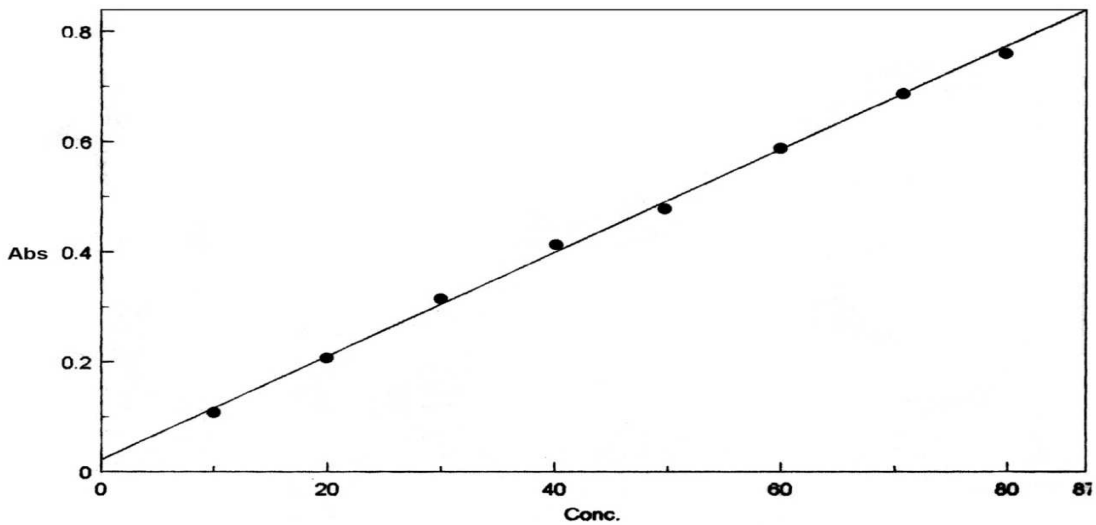


Figure 2: Calibration graph of Voglibose 282 nm

Preparation of sample solution

The proposed method was applied to analyte commercially available Voglibose tablet. Thirty tablets, each containing 0.2 mg of Voglibose were weighed and average weight was calculated. Quantity equivalent to 5 mg of Voglibose was weighed, transferred to a 100 ml volumetric flask, extracted and made upto volume with methanol and filtered through whatmann filter paper # 41. From this solution, suitable aliquots were prepared, then these dilutions were derivatized and scanned in UV region and absoprbances were noted at 282 nm and concentration was determined by linear regression equation.

Results and discussion

Linearity

The linearity of the response of the drug was verified at 2 to 100 µg/ml concentrations, but linearity was found to be between 10 -80 µg/ml concentrations. The calibration graphs were obtained by plotting the absorbances versus the concentration data and were treated by linear regression analysis. The equation of the calibration curve for Voglibose obtained Y = 0.0094x + 0.0226, the calibration curve was found to be linear in the aforementioned concentrations. The correlation coefficient (r^2) of determination was 0.9976. (Table 1, 2)

Table 1: Linearity of Voglibose

S. No.	Concentration (µg/ml)	Absorbance at 282 nm
1	10	0.1078
2	20	0.2074
3	30	0.3142
4	40	0.4158
5	50	0.4746
6	60	0.5889
7	70	0.7047
8	80	0.7560

Table 2: Optical and Regression Characteristics, Precision and Accuracy of the proposed method for Voglibose [21-23]

Parameter	voglibose
Absorption maxima	282 nm
Linearity Range (µg/ml)	10-80
Standard regression equation	Y = 0.0094x + 0.0226
Correlation Coefficient	0.997

Precision

Assay of method precision (intraday precision) was evaluated by carrying out six independent assays of test samples of Voglibose. The intermediate precision (interday precision) of the method was also evaluated using two different analysts, systems and different days in the same

laboratory. The relative standard deviation (RSD) and assay values obtained by two analysts were 0.28, 99.67 and 0.26, 99.68 respectively. (Table 3)

Table No.3- Determination of Precision

Sample number	Assay of Voglibose as % of labeled amount	
	Analyst-I (Intra-day precision)	Analyst-II (Inter-day precision)
1	99.42	99.70
2	99.63	99.23
3	99.58	99.57
4	99.10	99.88
5	100.12	99.98
6	99.20	99.25
Mean	99.50	99.60
RSD	0.36	0.31

Accuracy (Recovery test)[24]

Accuracy of the method was studied by recovery experiments. The recovery experiments were performed by adding known amounts of the drug in the placebo. The recovery was performed at two levels, 50 and 100% of Voglibose standard concentration . The recovery samples were prepared in before mentioned procedure. Three samples were prepared for each recovery level. The solutions were then analyzed, and the percentage recoveries were calculated from the calibration curve. The recovery values for Voglibose ranged from 99.7 to 101.5%. The result of analysis the recovery studies presented in table-4&5.

Table 4: Recovery Studies

Level	%Recovery	%RSD*
50%	99.7	0.2534
100%	101.5	0.3050

* RSD of six observations

Table 5: Analysis of formulation

Drug	Amount(mg/ tablet)		% label claim	%RSD*
	Labeled	Found		
Voglibose	0.2	0.194	97	0.1843

* RSD of six observations

The percentage recovery values indicates that there no interference from the excipients present in the formulation that developed method is found to be sensitive, accurate, precise and most reproducible. It can be used for the routine quality control analysis of Voglibose bulk drug too.

The proposed method for estimation of Voglibose in pharmaceutical formulation was found to be simple, accurate, economical and rapid. The interference of interfering component was neglected by selecting the proper λ_{max} for the component of interest.

Effect of time on stability of absorbance

The stability of the solution was checked by measuring the absorbance at regular intervals of time. It was observed that the absorbance remain stable for 10 hrs at room temperature and 48 hrs at refrigeration. The result of stability studies presented in table-6.

Table 6: Stability Studies

Concentration (µg/ml)	Absorbance of the Drug			
	Time in hrs	At room temperature	Time in hrs	Refrigeration
50	0	0.4746	0	0.4746
	5	0.4603	10	0.4651
	6	0.4413	15	0.4508
	7	0.4223	23	0.4318
	8	0.4081	26	0.4223
	9	0.3939	34	0.4081
	10	0.3749	48	0.3796

Conclusion

The results conformed the reproducibility, precision and accuracy of the method. The marketed formulations were analyzed by the proposed method and were found that there was no interference with the exicipents incorporated in the tablet formulation as seen from recovery studies. The method described can be used for the estimation of tablet formulations due to simplicity in preparation and cost effective. The results obtained all in close declaration and found to be satisfactory. The method can be adopted for the conformation of Voglibose in bulk as well as for its formulation.

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