IJBPAS, September, 2025, 14(9): 4794-4800

ISSN: 2277–4998

International Journal of Biology, Pharmacy and Allied Sciences (IJBPAS)

'A Bridge Between Laboratory and Reader'

www.ijbpas.com

A VALIDATED UV-SPECTROPHOTOMETRIC METHOD FOR VILDAGLIPTIN ASSAY USING GREEN SOLVENT

VARAPRASADA RAO K, SHANMUKHA RITHIKA D, SANDHYA M, MANASA P, RAJESH BABU KB*

Department of Pharmaceutical analysis, Vignan Institute of Pharmaceutical Technology, Visakhapatnam, Andhra Pradesh-530046, India

***Corresponding Author: Dr. Bhagavan Rajesh Babu Koppisetty: E Mail: koppisettybrbabu@gmail.com**

Received 15th March 2024; Revised 20th April 2024; Accepted 11th Aug. 2024; Available online 1st Sept. 2025

https://doi.org/10.31032/IJBPAS/2025/14.9.8810

ABSTRACT

This study primarily focuses on developing a novel UV method for the assay of vildagliptin in both pure form and pharmaceutical dosage forms. The process involves preparing standard and working solutions of vildagliptin, followed by the analysis of different concentrations of the working solution. The established method is then subjected to validation as per ICH guidelines. The results indicate that the developed method is sensitive and accurate, particularly within the concentration range of 4-24 µg/ml. The correlation coefficient (R2) was determined to be 0.999. Notably, there was no interference observed with the excipients present in the formulation. The proposed method holds potential for the analysis of vildagliptin in bulk and formulation, making it suitable for routine analysis

Keywords: Vildagliptin, UV-spectroscopy, validation. Anti-diabetic drug, spectrometry

INTRODUCTION:

Vildagliptin (VDG) is a once-daily dipeptidyl peptidase 4 (DPP-4) inhibitor used in the management of type 2 diabetes mellitus. VDG **(Figure 1)** is an orally active anti-hyperglycemic agent that selectively inhibits the dipeptidyl peptidase-4 (DPP-4) enzyme. It is used to manage

type II diabetes mellitus, where GLP-1 secretion and insulinotropic effects are impaired **[1]**. Its chemical formula $C_{17}H_{25}N_3O_2$ and IUPAC name is 1-[2-[(3-hydroxy-1-adamantyl) amino acetyl] pyrrolidine-2-carbonitrile. Molecular weight is 303.399g/mol. The primary mechanism of action of VDG exerts its blood glucose-lowering effects by selectively inhibiting the dipeptidyl peptidase-4 i.e, (DPP-4), which is an enzyme that, upon release from the intestinal cells, rapidly truncates and inactivates GLP-1 and GIP **[2]**. Oligopeptides are broken down by DPP-4 after the second amino acid from the N-terminal end. The half -lives of GLP-1 and GIP are markedly extended by the inhibition of DPP-4, resulting in higher quantities of active incretin hormones in the blood. VDG lowers fasting, postprandial and HbA1c levels of glucose. The alpha and beta cell glucose sensitivity is improved, and glucose dependent insulin secretion is also increased **[3]**.

Through a comprehensive examination of existing literature, it has been observed that only a small amount of research has been recorded on this topic regarding the assessment of VDG using HPLC and UV **[4-8]**. There are few spectrophotometric methods reported for analysis of VDG **[9-10]**. The newly established analytical technique used a green solvent water for method development and underwent validation in accordance with the ICH guidelines **[10-12]**. The primary objective of this study is to create straightforward, speedy, and cost-effective techniques for concurrently determining these medications within a tablet formulation.

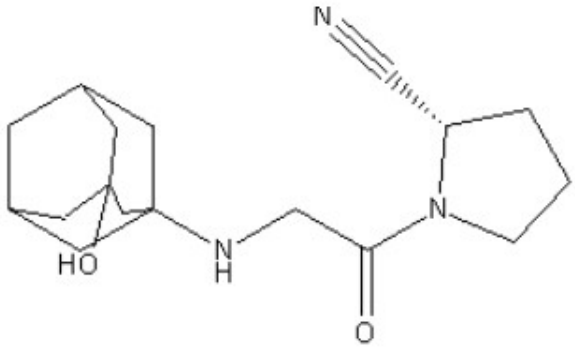

Figure 1: Structure of VDG

MATERIALS AND METHODS:

Instruments and Reagents:

A complimentary sample of VDG with a purity level of 99.98% was obtained from a manufacturing facility located in Visakhapatnam. The instruments employed in the study included UV/Visible spectrophotometer, Lab india, model T60 and analytical balance, Shimadzu, Japan. The investigation utilized analytical-grade

chemicals and reagents. VILDARAY branded VDG tablets, each containing 50 mg, were obtained for the formulation.

Standard stock solution (1000µg/ml):

A quantity of 100 mg of the drug was introduced into a 100 ml calibrated flask, where it was dissolved and topped up to the calibration mark with distilled water, resulting in 1000 µg/ml. This establishes the standard stock solution of VDG.

Working standard solution (100µg/ml):

A quantity of 2.5 ml was extracted from the standard stock solution mentioned earlier and transferred into a 25 ml calibrated flask. Distilled water was added to the flask to achieve a concentration of 100 µg/ml, and the solution was adjusted to the mark.

Construction of calibration curve:

Following that, it was subjected to scanning using a UV Spectrophotometer covering the 200-400 nm range, with distilled water employed as the blank. The peak absorbance was pinpointed at a wavelength of 206 nm. To generate different concentrations spanning from 4 to 24 µg/ml, portions were formulated using distilled water as the solvent. These samples were then assessed at the specified wavelength of 206 nm to ascertain their respective absorbance values. The collected data was subsequently used to construct a calibration curve.

RESULTS AND DISCUSSION

Method Validation:

Linearity:

Various samples of VDG were created within the 4-24 µg/ml range using the working standard solution (16 µg/ml). These solutions underwent scanning on a UV-spectrophotometer spanning the 200-400 nm range, with distilled water serving as the reference. The spectrum was captured at 206 nm **(Fig-2).** The data illustrated the relationship between concentration and absorbance, is depicted in **Table-1**. The results indicate a high degree of linearity in the established relationship.

Precision:

The method's precision was showcased through assessments of intra-day and inter-day variations. In the intra-day analysis, six separate solutions with 16 µg/ml were created and assessed twice daily, yielding a % RSD of 1.16. For the inter-day study, solutions of 16 µg/ml were formulated and was tested six times over two successive days, and the absorbance was noted (refer to **Table 2**). The calculated percentage of relative standard deviations was found to be below 2%.

Accuracy:

The method's accuracy was assessed using the standard addition method, wherein the percent

recovery of VDG was computed. Pre-quantified sample solutions of VDG were supplemented with known quantities of standard solutions at 80%, 100%, and 120% levels. These solutions were prepared in triplicate, and the accuracy, as indicated by the %recovery, was calculated and presented in **Table 3**. The %recovery was determined to be satisfactory.

Robustness:

The method's reliability was evaluated through the examination of a sample with a concentration of 16 µg/ml at three distinct wavelengths, including one at λmax, and recording the corresponding absorbance values. The outcomes presented in **Table 4** suggest that the method demonstrated robustness.

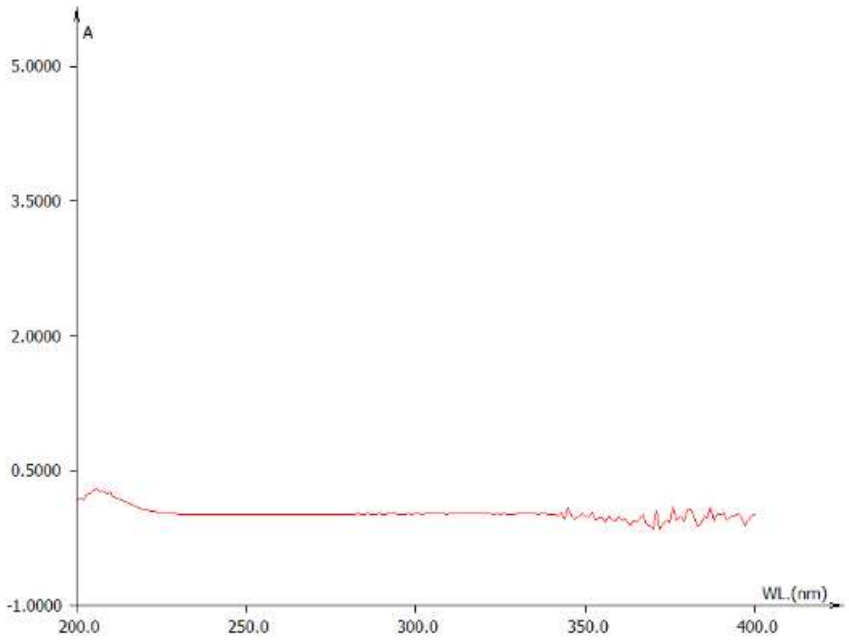

Figure 2: UV Spectrum of VDG

Table 1: Linearity Data

Conc. (µg/ml)	Absorbance
4	0.118
8	0.2812
12	0.4261
16	0.5852
20	0.7171
24	0.8837
Regression equation	y = 0.037x - 0.027
Correlation coefficient (R^2)	0.999

Table 2: Intermediate Precision

Conc. [µg/ml]	Absorbance	
	Analyst-1/Day-1	Analyst-2/Day-2
16	0.5828	0.5798
16	0.5765	0.5712
16	0.5898	0.5764
16	0.5722	0.5736
16	0.5822	0.568
16	0.5861	0.588
Mean	0.5816	0.576167
S.D	0.006382	0.007088
%RSD	1.097346	1.230284
%RSD (12 Determinations)	1.16	

Table 3: Accuracy of method

Level of addition	Formulation amount	Amount added	Theoretical amount	Experimental amount [n=3]	% recovery	% mean recovery ±SD
80%	16	12.8	14.4	14.22	98.79	98.7±0.005
100%	16	16	16	15.88	99.29	99.29±0.19
120%	16	19.2	17.6	17.48	99.31	99.3±0.22

Table 4: Robustness Study

Conc. (µg/ml)	Absorbance		
	222nm	223nm	224nm
16	0.5623	0.5828	0.5844
16	0.5722	0.5765	0.5985
16	0.5698	0.5898	0.5852
16	0.5632	0.5722	0.5985
16	0.5678	0.5822	0.5976
16	0.5612	0.5861	0.5998
AVG	0.566083	0.5816	0.594
SD	0.004487	0.006382	0.007165
%RSD	0.792571	1.097346	1.206262

Ruggedness:

To assess the ruggedness of the method, the sample was analyzed by two different analysts using the identical apparatus, and by the same examiner using two different cuvettes, with the respective absorbance values recorded. The results from the first analyst revealed a %RSD of 0.3546, while the second analyst showed a %RSD of 0.5476. These results indicate that the utilized methodology was robust, as no notable distinction is evident among various operators.

Sensitivity:

LOD and LOQ for the drug were determined through regression analysis from the calibration curve, resulting in values of 0.679 µg/ml and 2.059 µg/ml, respectively.

Assay of formulation*:*

The analysis of the obtained formulation involved assaying an equivalent weight of 25 mg of VDG formulation in a 25 ml calibrated

flask, utilizing distilled water as the diluent. The final concentration was adjusted to 16 µg/ml using distilled water. The assessment was conducted at a UV wavelength of 206 nm, revealing an assay result of 99.87%.

CONCLUSION:

The proposed method proved to be simple, exhibiting accuracy, precision, and robustness while being easily implementable. The calibration plot covered a broad range, and the recoveries of samples were consistent. The equipment and reagents utilized are likely to be accessible, even in basic laboratory setups. Therefore, the established method is recommended for regular use in quality control analysis of VDG. Additionally, it is deemed suitable for analyzing samples in accelerated stability studies, routine formulation analyses, and the assessment of drug substance.

REFERENCES:

[1] Mathieu C, Degrande E. Vildagliptin: a new oral treatment for type 2 diabetes mellitus. Vasc Health Risk Manag. 2008;4(6):1349-60.

[2] Keating GM. Vildagliptin: a review of its use in type 2 diabetes mellitus. Drugs. 2014 Apr;74(5):587-610.

[3] Moulinath Banerjee, Naveed Younis & Handrean Soran (2009) Vildagliptin in clinical practice: a review of literature, Expert Opinion on Pharmacotherapy, 10:16, 2745-2757.

[4] K. Hanumantha rao, A. Lakshmana rao and KB. Chandra sekhar-development and validation of HPLC method for the estimation of vildagliptin in pharmaceutical dosage form. International journal of pharmaceutical, chemical, and biological sciences. 2014,4 (2), 361-366.

[5] Rahima Khatun, Md Mirazunnabi. A validated reversed phase HPLC method for the determination of vildagliptin from tablet dosage form. International journal of pharmaceutical and life sciences. 2013, volume 2, 2305-0330.

[6] Ramesh jayaprakash, Senthil Kumar Natesan. Stability indicating RP-HPLC method development and validation for the simultaneous determination of vildagliptin and metformin in pharmaceutical dosage form. International journal of pharmacy and pharmaceutical sciences. 2017, volume 9, 0475- 1491.

[7] Pragati Ranjan Satpathy, V. Mohan Goud, Bhoga Bhagya, JVC. Sharma and N. Shyamala. Development and validation of RP-HPLC method for the

assay of vildagliptin. World journal of pharmacy and pharmaceutical sciences. 2014, volume 3, 2303-2310.

[8] Aparajita Malakar, Bishwajit Bokshi, Dilruba Nasrin. Development and validatin of RP-HPLC method of vildagliptin from tablet dosage form. International journal of pharmaceutical and life sciences. 2012, volume 1.

[9] Kumari B, Khansili A. Analytical Method Development and Validation of UV-visible Spectrophotometric Method for the Estimation of Vildagliptin in Gastric Medium. Drug Res (Stuttg). 2020 Sep;70(9):417-423. doi: 10.1055/a-1217-0296. Epub 2020 Aug 3. PMID: 32746479.

[10] Prasad PV, Divya K, Ramana MV, Krishnaveni A, Vinay J, Mahadeswar M. Analytical method development and validation for the estimation of vildagliptin in bulk and its dosage form using UV spectrophotometer. IAJPR. 2017;7(12):2231-6876.