EUROPEAN JOURNAL OF BIOMEDICAL AND PHARMACEUTICAL SCIENCES
http://www.ejbps.com

ISSN 2349-8870
Volume: 12
Issue: 7
Year: 2025

DEVELOPMENT AND VALIDATION OF UV SPECTROSCOPIC METHOD FOR ESTIMATION OF VILDAGLIPTIN IN TABLET DOSAGE FORM

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Article Received on 30/04/2025 **Article Revised on 21/05/2025** **Article Published on 11/06/2025**

ABSTRACT

Objective: To develop and validate simple, rapid, linear, accurate, precise and economical UV Spectroscopic method for estimation of Vildagliptin in tablet dosage form. **Methods:** The drug is freely soluble in Water. The drug was identified in terms of solubility studies and on the basis of melting point done on melting point apparatus of Equiptronics. It showed absorption maxima were determined in Water. The drug obeyed the Beer's law and showed good correlation of concentration with absorption which reflect in linearity. The UV spectroscopic method was developed for estimation of Vildagliptin in tablet dosage form and also validated as per ICH guidelines. **Results:** The drug is freely soluble in Water and Ethanol, Practically insoluble in ethyl acetate and insoluble in acetone. So, the Water is used as a diluent in method. The melting point of Vildagliptin was found to be 149-150˚C (uncorrected). It showed absorption maxima 230 nm in Water. On the basis of absorption spectrum the working concentration was set on 30 µg/ml (PPM). The linearity was observed between 10-50 µg/ml (PPM). The results of analysis were validated by recovery studies. The recovery was found to be 98.0, 99.0 and 98.2% for three levels respectively. The % RSD for precision and ruggedness was found to be 1.08% and 0.31% respectively. **Conclusion:** A simple, rapid, linear, accurate, precise and economical UV Spectroscopic method has been developed for estimation of Vildagliptin in tablet dosage form. The method could be considered for the determination of Vildagliptin in quality control laboratories.

KEYWORDS: Vildagliptin, UV Spectrophotometer, Melting Point, Assay Method, Validation, Accuracy, Linearity, Ruggedness, Precision.

INTRODUCTION

Vildagliptin, an antihyperglycemic drug, is having high water solubility and shorter elimination half-life. The administration of Vildagliptin results in rapid and near complete inhibition of DPP-4 activity. Vildagliptin shows weak inhibition of, and rapid dissociation from DPP-8 and DPP-compared to DPP-4.[1,2] This drug is a potent and selective inhibitor of dipeptidyl peptidase-IV (DPP-4). Oral vildagliptin was approved by the European Medicines Agency in 2008 for the treatment of type II diabetes mellitus in adults as monotherapy or in combination with metformin, a sulfonylurea, or a thiazolidinedione in patients with inadequate glycemic control following monotherapy.[3,4] It is marketed as Galvus It is a orally active drug and improves glycemic control in patients with type 2 diabetes (T2DM) by increasing pancreatic (α and β) islet function.[5] Thus vildagliptin suppress the inappropriate glucagon secretion seen in patients with T2DMand improves insulin secretion in them. It also reduces HbA1c when given as a single drug, without weight gain and with a little hypoglycemia, in combination with the other commonly prescribed classes of oral hypoglycemic drugs: a thiazolidinedione, a sulfonylurea or insulin.[6]

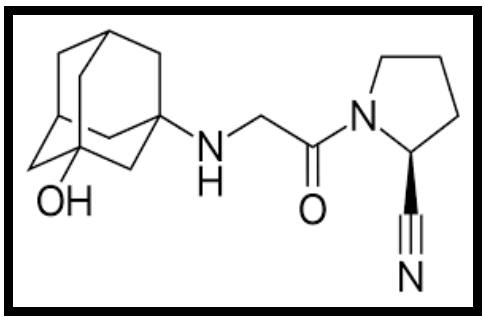

Fig. 1: Chemical Structure of Vildagliptin.

From literature review it's found that one method was reported on UV spectroscopic method for simultaneous estimation of Vildagliptin.[6] Also the method was reported on Stability indicating RP-HPLC method[7,8] on

Vildagliptin. Few methods were reported on Gas Chromatography-Mass Spectrometry[9] and by Tandem Mass Spectrometry[10] in Rat Plasma method development for Vildagliptin. But these methods have lack of accuracy and sensitivity. So, it needs to developed accurate, precise and sensitive UV method for the estimation and determination of Vildagliptin in tablet dosage forms.

MATERIALS AND METHODS

- **Instruments**

Shimadzu double beam UV-visible spectrophotometer 1700 Ultra with matched pair Quartz cells corresponding to 1 cm path length and spectral bandwidth of 1 nm, Bath sonicator and citizen weighing balance.
Melting point apparatus of Equiptronics were used.

- **Materials**

Vildagliptin was obtained as a gift sample. Vildagliptin tablets were procured from local pharmacy. Water was used throughout the experiment as a diluent. Freshly prepared solutions were employed.

Method development

A. Determination of ƛ max (10 PPM)[11,12]

50 mg weighed amount of Vildagliptin was dissolved into 100 ml of volumetric flask with Water. Pipette out 1 ml and added in 50 ml of volumetric flask dissolved and diluted up to the mark with Water. This solution was subjected to scanning between 200-400 nm and absorption maximum was determined.

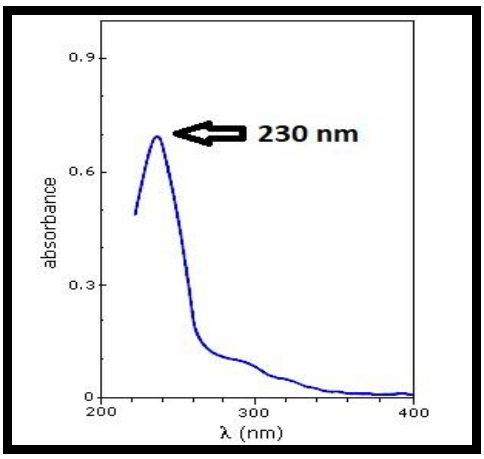

Fig. 2: Calibration Curve.

B. Preparation of Working concentration[12]

Preparation of Standard stock solution

Standard stock was prepared by dissolving 50 mg of Vildagliptin in 100 ml of Water to get concentration of 500 µg/ml (PPM).

Preparation of Standard solution

Pipette out 3 ml from standard stock solution and diluted up to 50 ml with Water to get concentration of 30 µg/ml (PPM).

C. Procedure for UV reading

Blank Solution: (For Auto zero)

Fill the cuvette with Water. Wipe it with tissue paper properly then placed inside the chamber. Note down the reading.

Standard Solution

Fill the cuvette with standard solution. Wipe it with tissue paper properly then placed inside the chamber. Note down the reading.

Sample Solution

Fill the cuvette with sample solution. Wipe it with tissue paper properly then placed inside the chamber. Note down the reading.

D. Procedure for sample preparations[11,12]

For analysis of commercial formulations; twenty tablets are taken weighed it and powdered. The powder equivalent to 50 mg of Vildagliptin was accurately weighed and transferred into the 100 ml of volumetric flask, added 60 ml Water, the solution was sonicated for 20 min. After sonication cool the flask and diluted upto 100 ml with Water. Filtered the solution through whatmann filter paper. Pipette out 3 ml of the above solution and diluted up to 50 ml with Water. The absorbance was measured at 230 nm. The absorbance was recorded:

Table 1: Absorbance of Dosage Form.

USV Pharmaceutical Limited Dovprela® 50 (50 mg)		
Sr. no.	**Sample**	**Absorbance**
1	Blank	0.0001
2	Standard	0.5567
3	Sample	0.5515

Table 2: Dosage Form Specifications.

Type	Company	M.D.	E.D.	Batch No.	Average weight (g)	Assay (%)
1	USV Pharma LTD Jalraa® 50	01/2025	02/2027	BYZ 543	0.118	99.07

E. Method of validation[11-16]

The proposed method was developed by using linearity, accuracy, precision and ruggedness as per ICH guidelines, 1996.

Linearity

The linearity of the proposed assay was studied in the concentration range 10 - 50 PPM at 230nm. The calibration data showed a linear relationship between concentrations.

Table 3: Linearity Studies.

Sr. no.	Sample Concentration	Absorbance
1	10 PPM	0.1859
2	20 PPM	0.3768
3	30 PPM	0.5517
4	40 PPM	0.7366
5	50 PPM	0.9295
Correlation coefficient		**0.999**

Accuracy

To ensure the accuracy of the method, recovery study was performed by preparing 3 sample solutions of 80, 100 and 120% of working concentration and adding a known amount of active drug to each sample solution and dissolved in 100ml of volumetric flask with Water and measuring the absorbance at 230nm.

Table 4: Accuracy Studies.

SPECTROPHOTOMETRIC METHOD			
Accuracy (%)	**Qty weighed (mg)**	**Qty found (mg)**	**Recovery (98-102%)**
80	0.8	0.78	97.5 ~ 98.0
100	1	0.98	99.0
120	1.2	1.18	98.17 ~ 98.2

Precision

The precision of the method was demonstrated by inter-day and intra-day variation studies. Five sample solutions were made and the % RSD was calculated.

Table 5: Precision studies.

Sr. No.	Sample Solution	Absorbance
1	Sample Solution 1	0.5585
2	Sample Solution 2	0.5515
3	Sample Solution 3	0.5521
4	Sample Solution 4	0.5474
5	Sample Solution 5	0.5425
MEAN		**0.5504**
SD		**0.0059**
% RSD		**1.0792 ~ 1.08**

Ruggedness

Ruggedness is a measure of the reproducibility of a test result under normal, expected operating condition from instrument to instrument and from analyst to analyst.

Table 6: Results for Ruggedness Studies.

Sr. No.	Analyst	Results	Mean	% Assay	% RSD
1	Analyst 1	0.5522 0.5534	0.5528	99.3	0.3055 ~ 0.31
2	Analyst 2	0.5549 0.5557	0.5553	99.73	

RESULTS

1. Solubility of Vildagliptin

Solubility test was passed as per criteria.

Table 7: Results for solubility studies.

Sr. no.	Title	Result
1	Water, Ethanol, DMSO	Freely Soluble
2	Ethyl Acetate	Practically Insoluble
3	Acetone	Insoluble

2. Melting point of Vildagliptin

The melting point of Vildagliptin was found to be 149-150˚C (uncorrected).

3. Results for linearity for assay method of Vildagliptin [Conc Vs Absorbance]

The linearity of method was determined at concentration level ranging from 10 to 50 µg/ml (PPM). The correlation coefficient value was found to be **(R^2) 0.999.**

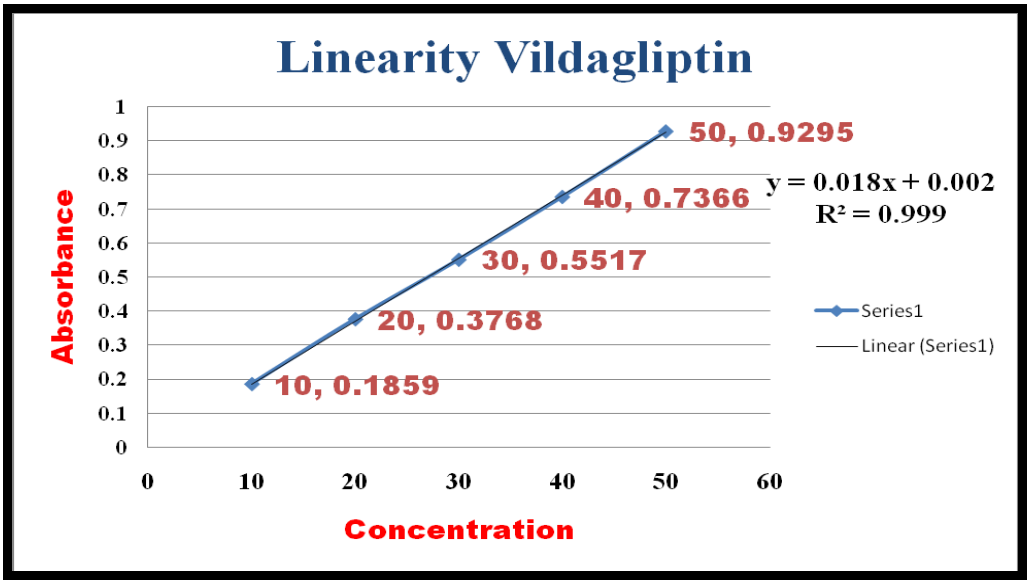

Fig. 3: Vildagliptin Standard Curve.

4. Results for accuracy for assay method of Vildagliptin

The accuracy of the method was determined by recovery experiments. The recovery studies were carried out and the percentage recovery were calculated and represented in Table - 4. The high percentage of recovery indicates that the proposed method is highly accurate. Accuracy results were found within acceptance criteria that are within 98-102%.

5. Results for precision for assay method of Vildagliptin

The % RSD for different sample of precision was found to be 1.08 and it is within acceptance criteria represented in Table - 5.

6. Results for ruggedness for assay method of Vildagliptin

The % RSD for different sample of ruggedness was found to be 0.31 and it is within acceptance criteria represented in Table - 6.

CONCLUSION

A method for the estimation of Vildagliptin in tablet form has been developed. From the spectrum of Vildagliptin, it was found that the maximum absorbance was 230 nm in Water. A good linear relationship was observed in the concentration range of 10-50 µg/ml (PPM). The high percentage recovery indicates high accuracy of the method. This demonstrates that the developed spectroscopic method is simple, linear, accurate, rugged and precise for the estimation of Vildagliptin in solid dosage forms. Hence, the method could be considered for the determination of Vildagliptin in quality control laboratories.

ABBREVARTIONS

1. PPM - Parts per Million
2. nm - Nanometer
3. TB - Tubelculosis
4. HPLC - High Performance Liquid Chromatography
5. UV - Ultra violet
6. FDA - Food and Drug Administration
7. DPP – Dipeptidyl Peptidase
8. T2DM - Type 2 Diabetes Mellitus
9. ICH - International Council for Harmonization
10. RSD - Relative Standard Deviation
11. SD - Standard Deviation
12. Qty - Quantity
13. C - Celsius
14. M.D. - Manufacturing Date
15. E.D. - Expiry Date

REFERENCES

1. en.wikepedia.org/wiki/Vildagliptin accessed on 02-06-2025.
2. *https://www.drugbank.ca/drugs/DB04876* accessed on 02-06-2025.
3. Ahrén BO, Landin-Olsson M, Jansson PA, Svensson M, Holmes D, Schweizer A. Inhibition of Dipeptidyl Peptidase-4 Reduces Glycemia, Sustains Insulin Levels, and Reduces Glucagon Levels In Type 2 Diabetes. The Journal of Clinical Endocrinology and Metabolism, 2004; 89(5): 2078-84.
4. Idris I, Donnelly R. Dipeptidyl peptidase-IV inhibitors: a major new class of oral antidiabetic drug. Diabetes Obes Metab, 2007; 9: 153–65.
5. Sunkara G, Sabo R, Wang Y, et al. Dose proportionality and the effect of food on vildagliptin, a novel dipeptidyl peptidase IV inhibitor, in healthy volunteers. J Clin Pharmacol, 2007; 47: 1152–8.
6. Naveed S, Rehman H, Qamar F, Zainab S. Method development and validation of Vildagliptin using UV spectrophotometer. International Journal of Pharmaceutical Science and Research, 2014; 5: 714-717.
7. Sultana R, Bachar SC, Rahman F. Development and validation of stability indicating assay method of

vildagliptin in bulk and tablet dosage form by RP-HPLC. International Journal of Pharmacy and Life Sciences, 2013; 4(4): 2530-4.
8. Barden AT, Salamon B, Schapoval EE, Steppe M. Stability-indicating RP-LC Method for the Determination of Vildagliptin and Mass Spectrometry detection for a main Degradation product. Journal of Chromatographic Science, 2012; 50(5): 426-32.
9. Uçaktürk E. Development of Sensitive and Specific Analysis of Vildagliptin in Pharmaceutical Formulation by Gas Chromatography- Mass Spectrometry. Journal of Analytical Methods in Chemistry, 2015; 2015: 1-7.
10. Attimarad M, Nagaraja SH, Aldhubiab BE, Nair AB, Venugopala KN. Determination on Vildagliptin in Rat Plasma by Capillary Electrophoresis Tandem Mass Spectrometry: It's Application to Pharmacokinetic Study. Indian Journal of Pharmaceutical Education and Research, 2017; 51(4): 636-643.
11. ICH draft Guidelines on Validation of Analytical Procedures: Definitions and Terminology, Federal Register, 60, IFPMA, Switzerland, 1995; 1260.
12. Beckeet. A.H, Stenlak. J.B, "Practical pharmaceutical chemistry edn 4th CBS Publisher & Distribution, New Delhi, 2004; 275-337.
13. British Pharmacopoeia. Volume I: published by the stationary office on behalf of the Medicine and Healthcare Products Regulatory Agencies, London, 2008; 76-77.
14. United States Pharmacopoeia. In Validation of Compendia Methods. 26th edn: Pharmacopoeia Convention Inc., Rockville, 2003; 2439–2442.
15. Indian Pharmacopoeia .Volume II. Ministry of Health and Family Welfare Government of India: Published by Indian Pharmacopoeia Commission, Ghaziabad, 2007; 692-693.
16. International Pharmacopeia. W.H.O. Volume 1, 3rd Edition, C.B.S. publishers and distributors, India, 1987; 130.