

Available online on 15.09.2019 at <http://jddtonline.info>

Journal of Drug Delivery and Therapeutics

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Research Article

A novel spectrophotometric method development and validation of Tenagliptin in its Tablet dosages form

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ABSTRACT

The aim of present work was to develop a new, economical & simple analytical method of Teneligliptin & its validation as per International Conference on Harmonization (ICH) guideline, which is a new antidiabetic drug that is used to treat Non-Insulin Dependent Diabetes Melitus (NIDDM) patients. A sensitive and specific Ultra Violet (UV) Visible method was developed & the analysis was carried out by using Shimadzu 1800 Spectrophotometer, 0.1(N) NaOH was used as a solvent. The UV- Visible Spectrophotometric estimation was carried out at a λ max of 242 nm using 1cm thick quartz cell. Validation of the newly developed method was performed as per International Conference on Harmonization (ICH) Q2R1 guideline in the following parameters: Calibration curve, Accuracy, Precision etc. Newly developed method obeys Beer's-Lambert's law in the concentration range of 5 - 25 μ g / ml, whereas correlation coefficient value is less than 1. The percent amount of drug estimated by this developed method was 100.91 %, which is very close to 100 %. The validation parameters like Accuracy, Precision, Linearity, Range, Limit of detection (LOD) & Limit of Quantification (LOQ) were studied for the developed method and was found to be within the limits.

Keywords: Teneligliptin, Validation, Method, Spectroscopy etc.

Article Info: Received 18 June 2019; Review Completed 10 Aug 2019; Accepted 26 Aug 2019; Available online 15 Sep 2019



Cite this article as:

Patra P, Ghosh S, Roy T, Ravi Kumar BVV, A novel spectrophotometric method development and validation of Tenagliptin in its Tablet dosages form, Journal of Drug Delivery and Therapeutics. 2019; 9(5):95-98
<http://dx.doi.org/10.22270/jddt.v9i5.3503>

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INTRODUCTION:

Analytical chemistry ¹ is an important branch of Chemistry. It is employed in separating, identifying and determining the relative amounts of the components making up a sample of matter. Distillation and precipitation techniques for the separation of components of complex mixtures prior to their qualitative or quantitative determination were replaced by sophisticated Chromatographic techniques ². Methods for separation and determination of chemical species are known collectively as instrumental methods of analysis. Most important instrumental methods like Spectroscopy, electrochemistry and Chromatography are used widely in present days ³.

The absorption spectroscopy is based on the measurement of transmittance **T** or the absorbance **A** of a sample solution filled in the transparent cell having a path-length of **l** cm. Generally absorbance is liner with the concentration **c** of the absorbing analyte, as represented by the following equation;

$$A = - \log T = kcl$$

Where, **k** is the absorptivity of the analyte. The photometric methods of analysis are based on the Bouger-Lambert-Beer's law, which establishes the absorbance of a solution is directly proportional to the concentration of the analyte. electronic excitation is found due to absorption of relatively high – energy radiation. ⁴⁻⁶.

Teneligliptin is an antidiabetic drug. It inhibits human dipeptidyl peptidase-4(DPP-4). By inhibiting it prevented the degradation of incretins GLP-1, GIP and promoted insulin release. The IUPAC name {(2S, 4S)- 4- [4- (3- methyl-1phenyl- 1Hpyrazol- 5-yl) piperazin- 1- yl] pyrrolidin- 2- yl} (1, 3-thiazolidin-3yl) methanone hemipentahydrobromide hydrate ⁷⁻⁸.

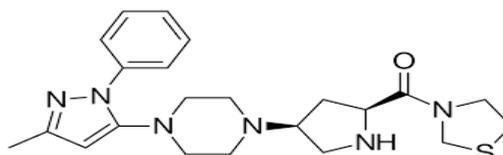


Figure 1. Shows structure of Teneligliptin

Literature review⁹⁻¹³ for Tenagliptin shows that, there are very few methods based on different techniques. However, there is no method reported for the detection of Teneligliptin alone, in bulk and Pharmaceutical formulation by UV - Visible Spectrophotometry. The aim of present work was to establish a very simple, sensitive, specific, Spectrophotometric method development and its validation for the detection of Tenagliptin in bulk drug and Pharmaceutical formulation.

MATERIALS AND METHODS:

Instrument:

A Shimadzu-1800 UV-Visible double beam Spectrophotometer with 1cm matched quartz cells was used for all spectral measurements. Authentic drug sample of Teneligliptine was given as a gift sample by Zydus Cadila, Ahmedabad. Tablets of Teneligliptin procured from local market.

Methods:

Solvents used for the determination of Teneligliptin in UV Spectrophotometer:

Uv - Visible Spectrophotometric method which involves the determination of Teneligliptine in Pharmaceutical formulation and has an absorption maximum at 244 nm in 0.1 N NaOH. It obeys beer's law in concentration range 5 - 25 µg/ml.

Solvent selection:

In order to select suitable solvent for determination of Teneligliptine, various solvents were selected for the solubility studies and it was found that Teneligliptine was freely soluble in the following solvents: water, ethanol, methanol, 0.1N NaOH, Acetonitrile, DMSO (Di Methyl Sulfa Oxide) etc. In the present investigation 0.1 (N) NaOH was used for initial dilution and all the remaining dilutions also carried out by Water only.

Preparation of standard stock solution:

It was prepared by dissolving accurately weighed 50 mg of Teneligliptine in 50 ml of 0.1N NaOH in 50 ml volumetric flask (stock solution-1, 1000 µg/ml). 1ml of stock solution-1 was diluted to 10 ml with Water (stock solution-2, 100 µg/ml), then 1ml of stock solution-2 was diluted to 10 ml with Water (stock solution-3, 10 µg/ml). Then absorbance of resulting solution was measured against respective blank solution in the UV region of 200-400 nm, which shows maximum absorbance at 242 nm.

Preparation of standard curve:

Aliquots of standard solution - 2 of Teneligliptine ranging from 0.5 ml - 2.5 ml were transferred into a series of 10 ml volumetric flasks separately to prepare concentration ranges from 5 µg/ml to 25 µg/ml. The volume in each flask was made up to 10 ml with water and the absorbance were measured at 242 nm against solvent blank and the absorbance values. The obtained absorbance values are plotted against the concentration of Teneligliptine to get the calibration graph. The concentration of the unknown sample was determined from the calibration graph. The correlation coefficient was determined.

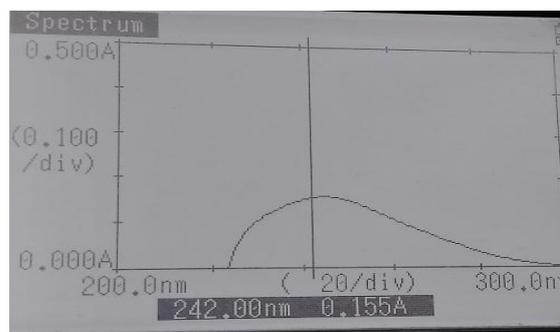


Figure 2. Shows UV Spectrum of Teneligliptin

Sample preparation of Teneligliptin:

Taken 10 tablets, powdered and weighed accurately equivalent to 50 mg of Teneligliptin was taken to a 50 ml cleaned and dried volumetric flask. This was then diluted with 0.1 N NaOH solution upto 50 ml. Then taken 1ml of sample solution and dilute with Water, volume was made up to 10 ml (100 µg/ml). This was marked and labeled as Stock solution. Further, an amount of 1ml was pipetted out from the above stock solution into a 10 ml volumetric flask and diluted up to the mark with diluent to get 10 µg/ml.

Method validation:

Various methods for analysis of Tenagliptin in bulk and Pharmaceutical formulation were carried out. Then the developed method was validated in the following parameters as ICH Q2B guideline

Accuracy:

Accuracy is the closeness if the test results obtained by the method to the true value. To study the accuracy, 10 tablets of Teneligliptin were taken, powdered the tablets used to carry out the analysis. Recovery studies were carried out by addition of standard solution (5, 10, 15 µg/ml) to the sample at 3 different concentration levels.

Precision:

Accurately weighed amount of 50 mg of Teneligliptin was taken to a 50 ml cleaned and dried volumetric flask. This was then dissolved with 0.1 N NaOH up to 50 ml. Then 1ml of sample solution was taken and dilute with Water, volume was made up to 10 ml (100 µg/ml). Further, an amount of 1ml was pipetted out from the above stock solution into a 10 ml volumetric flask and diluted up to the mark with diluent to get 10 µg/ml. Then 6 replicates of 10 µg/ml solutions was prepared in the same manner.

Linearity:

Five point Linearity curve was generated with the appropriate volumes of the working standard solutions for UV methods.

Limit of Detection:

The Limit of detection is the lowest concentration of the analyte in a sample that can be detected but not necessarily determined in a quantitative fashion using a specific method under the required experimental conditions. Such a limit is expressed in terms of a concentration of analyte (example: µg / lit) in the sample.

LOD = 3.3 SD / S where, SD - is the Standard Deviation, 'S' - is the slope of the calibration curve for analyte.

Limit of Quantification:

The LOQ is the lowest concentration of analyte in a sample that may be measured with an acceptable level of accuracy and precision under the above mentioned conditions of the method. LOQ value varies with the method used & the nature of sample.

LOQ = 10 SD / S, where, SD – is the Standard Deviation, ‘S’ - is the slope of the calibration curve for analyte.

RESULTS AND DISCUSSION:

The development of a simple, rapid, sensitive and accurate analytical method for the routine quantitative determination

of samples will reduce unnecessary tedious sample preparations and the cost of materials and labour. Specific chromophore present in the structure of Tenapliptin molecule, absorb at a particular wavelength of UV region and this fact was successfully employed for their quantitative determinations using the UV – Visible Spectrophotometric method. The absorption maxima (λ_{max}) of the drug was determined by taking scans of the drug sample solutions in the entire UV region. It was found to be that only one peak was observed in this method at the wavelength of 242 nm (λ_{max}).

Accuracy:

Table 1: Accuracy Data of Teneligliptin

%CONC	Absorbance		CONC. (µg/ml)	%Purity	%Recovery	%Mean
	Standard solution	Sample solution				
50%	0.093	0.099	5	106.7		
50%	0.090	0.097	5	107.8	106.63	
50%	0.091	0.096	5	105.4		
100%	0.290	0.285	10	98.2		101.91
100%	0.270	0.265	10	98.1	98.3	
100%	0.287	0.283	10	98.6		
150%	0.326	0.330	15	101.2		
150%	0.332	0.328	15	98.8	100.8	
150%	0.329	0.337	15	102.4		

Acceptance Criteria:

The % Recovery should be between 98.0 to 102.0% in each level.

The results obtained for recovery are within the limits.

So, it is observed from the above table that, accuracy of the newly developed method is 101.91%, which is mentioned in table 1. Hence the method is accurate.

Precision:

Table 2: Precision Data of Teneligliptin

Concentration (µg/ml)	Absorbance
10	0.240
10	0.245
10	0.245
10	0.247
10	0.248
10	0.246
Mean	0.245
S.D	0.00279
% RSD	1.13

Acceptance criteria:

% RSD of different analytes should not be more than 2. The % RSD value of the newly developed method is 1.13, as mentioned in Table 2. Which is within the limit, hence the method is precise.

Linearity & Range:

Table No 3: Linearity Table of Teneligliptin

Concentration(µg / ml)	Absorbance
5	0.134
10	0.240
15	0.342
20	0.444
25	0.547

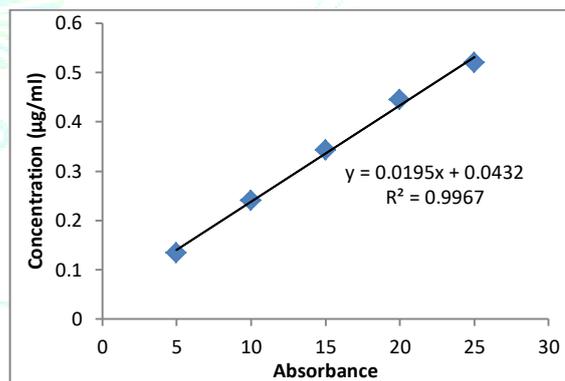


Figure 3. Linearity Graph of Teneligliptin

Acceptance criteria:

Correlation coefficient (R^2) should not be less than 0.999

The correlation coefficient obtained was 0.996 in the newly developed method, as per Table 3. which is very near to acceptance limit. The linearity was established in the range of 5% to 25% for Teneligliptin, which is mentioned in Fig. 3.

LOD & LOQ:

The LOD & LOQ were calculated as per the formulas, mentioned above & was reported as follows.

Table No 4: LOD & LOQ Table

Sample	LOD (µg/ml)	LOQ (µg/ml)
Teneligliptin	0.42	1.42

CONCLUSION:

A simple, economic, accurate and precise UV - Vis Spectrophotometric method was successfully developed. The method developed was statistically validated in terms of Accuracy, Linearity, Precision, LOD, LOQ. Statistical result of all the validation parameter was within the prescribed limits. Solvent is very economic & very easy to prepare. Hence, the UV Spectrophotometric method developed for Tenueligliptin in Tablet dosages form was found to be economic, rapid, simple, sensitive, precise, and accurate.

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