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Research Article

A NOVEL SPECTROSCOPIC METHOD FOR THE SIMULTANEOUS DETERMINATION OF PREGABILIN AND VILDAGLIPTIN IN SYNTHETIC MIXTURE

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ABSTRACT

The present study was simultaneous determination of Pregabalin and Vildagliptin in synthetic mixture. The wave length of Pregabalin and Vildagliptin are measured at 217 nm and 204 nm using 0.2M Hcl. Results showed that the two drugs obey beer's law with concentration ranges of 60-100 µg/ml for Pregabalin and concentration range of 1-10 µg/ml for Vildagliptin, respectively. The relative standard deviation of both intra-day and inter-day precision were less than 2% found. The LOD and LOQ were found to be 1.2 µg/ml and 3.69 µg/ml for Pregabalin and 1.06 µg/ml and 3.19 µg/ml for Vildagliptin, respectively. The recovery of Pregabalin and Vildagliptin in synthetic form was found within the limits. Hence the present method was successfully adopted for the simultaneous estimation of Pregabalin and Vildagliptin in bulk sample preparations.

Keywords: Pregabalin, Vildagliptin, Determination, Validation.

INTRODUCTION

Pregabalin is chemically (S) -3-(amino methyl)-5-methylhexanoic acid ,a potent gabapentinoid and structural analogue of the neurotransmitter GABA, as well as of GABOB which had an ion channel modulator with rapid analgesic, anticonvulsant and anxiolytic effects[1-2]. Pregabalin was used for the seizures, nerve damage, neuropathic pain and diabetics. Vildagliptin is chemically (2S)-1-[N-(3-Hydroxy-adamanatan-1-yl) glycyl]-2-pyrrolidinecarbonitrile and it is an anti diabetic agent belongs to the dipeptidyl peptidase IV (DPP-4) inhibitor[3]. Vildagliptin is used for the treatment for the hyperglycemia in type 2 diabetes mellitus. The literature survey gives the information about UV[4-7] and HPLC[8-10] methods for the simultaneous estimation and individual estimation with other drugs of Pregabalin and Vildagliptin.till date there is No method reported for the simultaneous determination of Pregabalin and Vildagliptin in synthetic mixture. The aim of the present study was to develop and validate a simple, sensitive, precise, new method for the simultaneous determination of Pregabalin and Vildagliptin in synthetic mixture.

MATERIALS AND METHODS

Instruments and Apparatus: ElicoSL164 UV-Visible spectrophotometer having double beam detector configuration.

Reagents and Chemicals: Pregabalin and Vildagliptin are obtained from Spectrum Labs, Hyderabad as gift samples with 99.99 % w/w assay values and without further purification. The absorption spectra of reference and test solution were carried out in a 1 cm quartz cell over the range of 200-400 nm. All chemicals of analytical grade are used.

Selection of Solvent: 0.2 M hydrochloric acid was used throughout the study.

Selection of Method and Wave Length

UV scan range of 200 nm to 800 nm was selected for the proposed method of Pregabalin and Vildagliptin. The wavelength corresponding to maximum absorbance was found at 217 nm for Pregabalin and 204 nm for Vildagliptin, respectively. The intercept of calibration line of the drug was determined by linear regression analysis.

Preparation of Pregabalin standard stock solution

Pregabalin pure drug 100 mg was weighed and dissolved in 100 ml of 0.2 M Hcl. 10 ml of the solution was taken from above solution and then make up to 100 ml with 0.2 M Hcl. 10 ml of the solution was separated from second stock solution and make up to 100 ml with 0.2 M Hcl and the obtained concentration is 10 mcg/ml. The absorbance is measured for samples and blank at 200-400 nm.

Preparation of Vildagliptin standard stock solution

Vildagliptin pure drug 100 mg was weighed and dissolved in 100 ml of 0.2 M Hcl. 10 ml of the solution was taken from above solution and then make up to 100 ml with 0.2 M Hcl. 10 ml of solution was separated from second stock solution and make up to 100 ml with 0.2 M Hcl and the concentration would be 10 mcg/ml. The absorbance is measured for samples and blank at 200-400 nm.

Preparation of Pregabalin sample stock solution

Weigh 20 tablets and powdered with the help of mortar and pestle, then weigh accurately a quantity of the powdered tablet equivalent to 0.025 gm of Pregabalin. Extract the drug with three portions of 20-25 ml of 0.2 M Hcl, filter through whatsmann filter paper. Combine all the three filtrate and make up the volume up to 100 ml with 0.2 M Hcl, then took 10 ml and dilute to 100 ml with 0.2 M Hcl. To get the resulting solution, took 10 ml of above solutions and dilute to 100 ml with 0.2 M Hcl. Measure the absorbance of standard and sample at 217 nm and deduce the results by comparison.

Preparation of Vildagliptin sample stock solution

The entire residue left after the extraction of Pregabalin as described above is used for estimation of Vildagliptin. Transfer the residue left on the filter paper to 100 ml volumetric flask with the help of 0.2 M Hcl. Make up the volume up to 100 ml with 0.2 M Hcl. Take 10 ml of this solution of Vildagliptin and dilute to 100 ml with 0.2 M Hcl. To get final concentration, took 10 ml of above solutions and dilute to 100 ml with0.2 M Hcl. Measure the absorbance of standard and sample at 204 nm and deduce the results by comparison.

Determination of λ Max

UV scan range of 200 nm to 800 nm was selected to determine maximum absorbance of Pregabalin and Vildagliptin by using 10 µg/mL solutions, the wave length corresponding to maximum absorbance was found at 217 nm for Pregabalin and found at 204 nm of Vildagliptin, respectively. The λ max values are shown in Figure 3 and Figure 4.

Method validation[11]**:** The spectroscopic simultaneous estimation of Pregabalin and Vildagliptin is validated as per the guidelines of ICH to determine the statistical parameters like linearity, precision, accuracy, LOD and LOQ of the proposed method.

Linearity plot: Standard stock solutions of Pregabalin and Vildagliptin in appropriate dilutions as per the proposed method. According to beer-lambert's law the concentration range of Pregabalin found to be 60-100 µg/mL and concentration range of Vildagliptin found to be 10-100 µg/mL, So that linearity in Figure.5 and Figure.6 are linear in the given concentration range.

Precision: The precision of the proposed method of Pregabalin and Vildagliptin were estimated by using concentrations of the drugs were analyzed six times in a day (intra-day precision) and six continuous days (inter-day precision). Data is given in Table 4.

Accuracy: The accuracy of the proposed method of Pregabalin and Vildagliptin were estimated by using standard addition method. This process is carried out by adding different amounts namely 50%, 100% and 150% of the pure samples to be pre-analysed formulation. Accuracy data of the drugs was shown in the Table 5 and 6.

LOD and LOQ: LOD is Limit of Detection and LOQ is Limit of Quantitation. The LOD and LOQ of Pregabalin and Vildagliptin were determined (Table 1) by using standard deviation of the response and slope approach as per the directions of International Conference on Harmonization (ICH) guidelines. The limits of detection (LOD) is calculated by using the equation LOD =3sk Where, S = intercept of the standard deviation K = the slope of the calibration curve (mean), the limits of quantitation (LOQ), is calculated by using the equation LOQ =10 SK Where, S = intercept of the standard deviation K = the slope of the calibration curve (mean).

Table 1: Optical Parameters of Pregabalin and Vildagliptin

S. No	Parameters	Pregabalin	Vildagliptin
1	Absorption maxima(nm)	217	204
2	Linearity Range(mcg/ml)	60-100	10-100
3	Standard Regression Equation	Y=0.0096X - 0.0053	Y=0.0045X - 0.0009
4	Correlation Coefficient (r^2)	0.9996	0.9998
5	Molar extinction coefficient	0.0083	0.0411
6	Accuracy (%recovery ±SD)	98.78± 0.038	99.45± 0.021
7	Precision	99.03% (Intra-day precision)and 99.04% (Inter-day precision)	98.74% (Intra-day precision) and 98.80% (Inter-day precision)
8	Sandell's Sensitivity (mg/cm2/0.001absorbance unit)	0.12	0.024
9	LOD (mg/ml)	1.21	1.06
10	LOQ (mg/ml)	3.69	3.19

Table 2: Linearity data of Pregabalin

S.NO	Concentration (mcg/ml)	Absorbance
1	60	0.560
2	70	0.660
3	80	0.760
4	90	0.860
5	100	0.960

Table 3: Linearity data of Vildagliptin

S. No	Concentration	Absorbance
0	0	0
1	10	0.045
2	20	0.090
3	30	0.135
4	40	0.183
5	50	0.231
6	60	0.276
7	70	0.320
8	80	0.362
9	90	0.405
10	100	0.452

Table 4: Precision data of Pregabalin

Sample number	Assay of Pregabalin	Assay of Pregabalin
	Intra-day precision	Inter-day precision
1	99.00	99.00
2	99.06	99.06
3	99.02	99.07
4	99.03	99.08
5	99.04	99.09
6	99.05	98.99
Mean	99.03	99.04
%RSD	0.022	0.044

Table 5: Precision data of Vildagliptin

Sample number	Assay of Vildagliptin	Assay of Vildagliptin
	Intra-day precision	Inter-day precision
1	98.71	98.79
2	98.73	98.80
3	98.75	98.83
4	98.78	98.81
5	98.72	98.77
6	98.77	98.84
Mean	98.74	98.80
% RSD	0.028	0.026

Table 6: Accuracy data of Pregabalin

Ingredient	Tablet amount (mg/ml)	Level of addition (%)	Amount added (mg)	Drug found (mg/ml)	% Recovery	Average % recovery
Pregabalin	100	50	5	4.89	97.80	
	100	100	10	9.87	98.70	98.78± 0.038
	100	150	15	14.98	99.86	

Table 7: Accuracy data of Vildagliptin

Ingredient	Tablet amount (mg/ml)	Level of addition (%)	Amount added (mg)	Drug found (mg/ml)	% Recovery	Average % recovery
Vildagliptin	100	50	5	4.97	99.40	
	100	100	10	9.93	99.30	99.45 ± 0.021
	100	150	15	14.95	99.66	

Table 8: Assay data

Formulation	Drug Name	Label claim (mg)	Amount found (mg)	% Amount found
Tablet-1	Pregabalin	100	99.95	99.95
	Vildagliptin	100	99.89	99.89

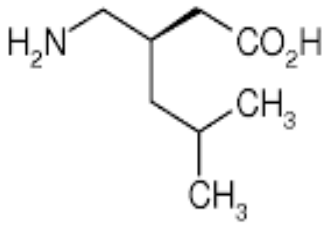

Figure 1: Chemical structure of Pregabalin

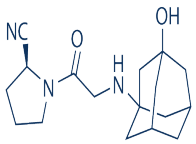

Figure 2: Chemical structure of Vildagliptin

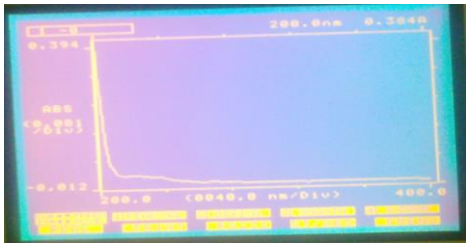

Figure 3: Determination of λ-max of Pregabalin at 217 nm

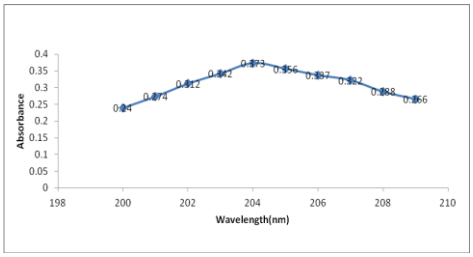

Figure 4: Determination of λ-max of Vildagliptin at 204 nm

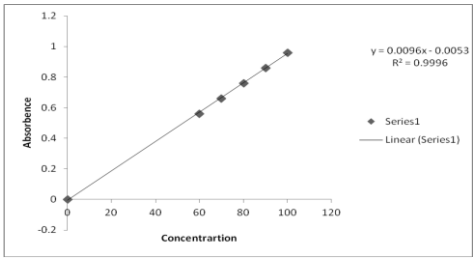

Figure 5: Linearity plot of Pregabalin (60-100 mcg/ml)

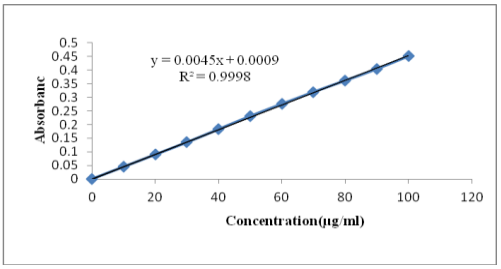

Figure 6: Linearity plot of Vildagliptin (10-100 mcg/ml)

RESULTS AND DISCUSSION

For the selection of analytical wavelength, Pregabalin and Vildagliptin solution were prepared separately by appropriate dilutions of standard stock solution and scanned in the spectrum mode from 200-400 nm by ELICO Double beam SL 264 UV/VIS spectrophotometer. The λ_{max} of 217 nm and 204 nm were selected for the simultaneous determination of Pregabalin and Vildagliptin and the absorption maxima curves were shown in Figure.3 and Figure.4. The linearity plot for Pregabalin and Vildagliptin prepared in the concentration range of 60-100 µg/mL and 10-100 µg/mL, respectively. The proposed method obeyed Beer's law in the concentration range of 10-100 µg/mL with a good correlation coefficient of $r^2 = 0.9996$ for Pregabalin and beer's law in the concentration range of 1-10 µg/mL with a good correlation coefficient of $r^2 = 0.9991$ for Vildagliptin, respectively. The optical characteristics and validation parameters of the proposed analytical methods were represented in Table 1. The precision of the method with intra-day and inter-day precision was found to be good with % RSD less than 2 which indicate that the method was precise and the results presented in Table 4 and 5. The recovery study results were found to be in the range of 97.80% to 99.86 % for Pregabalin and 99.30% to 99.66% for Vildagliptin, percentage RSD less than 2, indicates that method was accurate. Results of accuracy were enclosed in Table 6 and 7. The LOD and LOQ were found to be 1.21 µg/mL and 3.69 µg/mL, 1.06 µg/mL and 3.19 µg/mL for Pregabalin and Vildagliptin, respectively. The LOD and LOQ results shown in Table 7. The developed method was eventually utilized in the analysis of tablet formulation and was found to be within the proposed limits and the mean % assay values. tablet formulations were found to be 98.78 ± 0.038% for Pregabalin and 99.45 ± 0.021% for Vildagliptin. The assay results shown in Table 8. No interference was found in the spectrogram of the formulation within the absorbance indicating that excipients used in tablet formulation did not interfere with the simultaneous estimation of the Pregabalin and Vildagliptin by the proposed UVspectroscopic method.

CONCLUSION

The present paper reveals a simple, sensitive, precise, low cost and a novel spectroscopic method for the simultaneous determination of Pregabalin and Vildagliptin in synthetic mixture, which has been developed and validated as per the guidelines of International conference on Harmonisation.

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