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Analytical Method Development and Validation for Piracetam as Bulk and in Pharmaceutical Formulation

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Abstract: A new, simple, rapid and reliable UV Spectrophotometry method was developed and validated for the estimation of piracetam in bulk and in pharmaceutical formulation. Chemically, piracetam is 2-oxo-1-pyrrolidine acetamide, a nootropic drug used to enhance cognition and memory, slow down brain aging, increases blood flow and oxygen to the brain, aid stroke recovery, and improves Alzheimer's, Down Syndrome, dementia, and dyslexia. The technique was applied using methanol as diluent. The first order derivative spectra were obtained at n=5, $\Delta\lambda$=2.0 nm, and determinations were made at 214 nm. The method showed high specificity in the presence of formulation excipients and good linearity in the concentration range of 10-80 µg/ml. The intra and interday precision data demonstrated the method has good reproducibility. Accuracy was also evaluated and results were satisfactory (mean recovery of 99.35%). The method was found to be accurate, precise and reproducible and can be used in routine determination of piracetam as bulk and in formulations. The method was validated according to ICH guidelines and its updated convention. This method is also fast and economical in comparison to the more time consuming HPLC method regularly used for formulation screening and quality control and can be used routinely by any laboratory possessing a spectrophotometer with a derivative accessory.

Keywords: Derivative spectroscopy, UV Spectrophotometer, Piracetam, Validation, Formulation.

INTRODUCTION

Piracetam is a nootropic drug It is a drug which is claimed to enhance cognition and memory, slow down brain aging, increase blood flow and oxygen to the brain, aid stroke recovery, and improve Alzheimer's, Down syndrome, dementia, and dyslexia, among others.[1]Piracetam's chemical name is 2-oxo-1-pyrrolidine acetamide. Literature survey revealed HPLC methods were developed for the estimation of piracetam in biological fluids.[2,6,7,8,11] Capillary electrophoresis[3], thin layer densitometric determination[5], micellar electrokinetic chromatography[9] methods were also developed for the estimation of piracetam in biological fluids. There are no methods reported for the analysis of piracetam in bulk or pharmaceutical formulation. Derivative spectrophotometry is an analytical technique of great utility for extracting both qualitative and quantitative information from spectra composed of unresolved bands, and for eliminating the effect of baseline shifts and baseline tilts. It consists of calculating and plotting one of the mathematical derivatives of a spectral curve. Derivative spectrophotometry is now a reasonably prized standard feature of modern micro computerized UV spectrophotometry [12]. The objective of the present study was to develop a simple, economical and

accurate analytical method for the estimation of piracetam in bulk as well as pharmaceutical dosage forms.

EXPERIMENTAL

Instrument

A Jasco UV recording spectrophotometer (Model V-530) was employed with spectral bandwidth (resolution) of 2 nm and data pitch of 0.5 nm with automatic wavelength correction and a pair of 10 mm quartz cells, high Precision Electronic Weighing Balance : Contech Make Model CA-224, Ultrasonic bath.

Reagent

Methanol (A.R.)

Procedure

Method of Analysis

The standard stock solution was prepared by dissolving 10 mg of piracetam in 100 ml methanol. Finally standard stock solution of 10 µg/ml was prepared by diluting 1 ml of the above solution to 10 ml with methanol.

The UV scan of this sample is then taken which does not show distinctive peak or trough. Further, first derivative spectra in the same range shows a good peak at λ_{max} 214 nm (Fig.1). Pure standard drug solution in methanol was used to plot calibration curve. The curve shows linearity in the concentration range of 10-80 µg/ml for piracetam at D_1 214nm.

Analysis of tablet formulation

For analyzing tablet formulations, 10 tablets of piracetam were accurately weighed and powdered. An amount of tablet triturate equivalent to label claim of piracetam was weighed and transferred in 100 ml calibrated volumetric flask, diluted with methanol stirred for about 30 min and then volume made up with methanol. This solution was filtered to remove any insoluble matter. 1 ml of the filtrate was diluted to 100 ml. 5 ml of the above solution was diluted to 10 ml with methanol to get a final concentration of 40 µg/ml and is measured at D_1 214 nm. The concentrations of drug in samples were determined using calibration curve.

RESULTS AND DISCUSSION

Under the experimental conditions described, the graph obtained for first derivative spectra showed linear relationship. Regression analysis using the method of least-squares was made for the slope, intercept and correlation coefficient values. The regression equations of calibration curves was $y = 1.32 \times 10^{-2} x + 9.54 \times 10^{-2}$, ($r = 0.9989$). The range was found to be 10-80 µg/ml.

To study the accuracy of the proposed methods, and to check the interference from excipients used in the dosage forms, recovery experiments were carried out by the standard addition method. The recovery studies were carried out by the addition of pure drug to preanalysed solution of sample. It was carried out at three different level i.e. 80,100 and 120% and was performed by adding known amount of standard drug solutions of piracetam to preanalysed tablet solutions. The resulting solutions were then reanalyzed by proposed method. The results of recovery studies are shown in Table 1.The mean recovery was found to be 99.15%.

Precision should be investigated using homogeneous, authentic samples. However, if it is not possible to obtain a homogeneous sample it may be investigated using artificially prepared samples or a sample solution. The precision of an analytical procedure is usually expressed as the variance, standard deviation or coefficient of variation of a series of measurements. (Table 2). Low values of %RSD are indicative of high precision of the methods. The repeatability and ruggedness study signifies the reproducibility of the method. (Table 3)

The developed derivative spectrophotometric method was found to be simple, accurate, precise and economical method. It is worthy to mention that no expensive or toxic solvents or reagents are used.Based on the validation study data, it can be concluded that the proposed method is accurate and precise for the analysis of piracetam. No interference was found from excipients used in tablet formulation and hence the method is suitable for analysis of piracetam in bulk and in tablet formulation in a routine manner.

Table 1: Results of Recovery Studies

Sr. No.	Amount of Drug Added (µg/mL)	%Recovery* ± SD	% R.S.D
1 .	32	99.58 ± 0.41	0.43
2.	40	99.1 ± 0.60	0.64
3.	48	98.8 ± 0.88	0.89

*mean of three estimations at each level

Table 2: Statistical Data for Analysis

Parameters	Laboratory Sample	Commercial Formulation
Mean % estimation	99.45	99.93
Standard Deviation	0.512	0.485
Standard error of mean	0.224	0.214
Coefficient of Variation	0.544	0.485

Table 3: Results of Repeatability and Ruggedness studies

Parameters	% RSD
Precision %RSD	
Intra-day (n = 3)	0.40 - 1.46
Inter-day (n = 3)	0.72 - 1.39
Repeatability(n=6)	0.89
Ruggedness (n=5)	
Analyst I	0.65
Analyst II	0.74

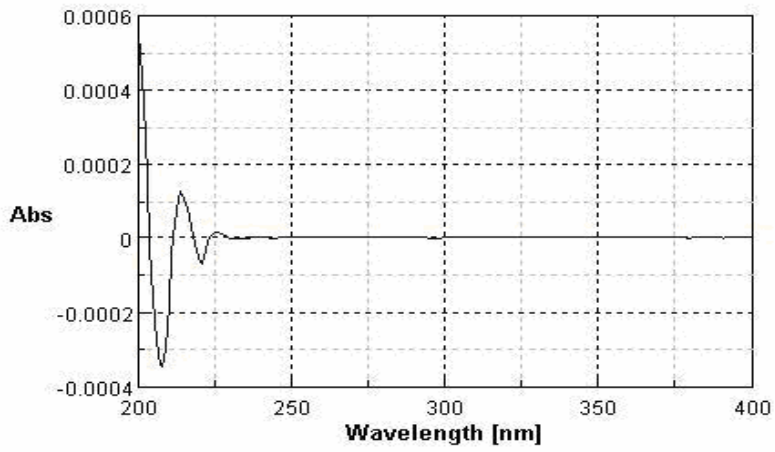

Figure 1 First derivative spectra of Piracetam

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