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Development and validation of new analytical method for the estimation of fluoxetine in bulk and dosage form by UV spectrophotometry

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ABSTRACT

A simple, rapid and precise method is developed for the quantitative determination of Fluoxetine in combined pharmaceutical-dosage forms. The method was based on UV Spectrophotometric determination of Fluoxetine drug using Beer-Lamberts Law. It involves absorbance measurement at 224 nm (λmax of Fluoxetine) in water. For UV Spectrophotometric method, linearity was obtained in concentration range of 5-30 mcg/ml with regression 0.999 for Fluoxetine respectively. Recovery was in the range of 98 -102%; the value of standard deviation and %R.S.D was found to be < 2 shows high precision of the method.

Keywords: Fluoxetine; UV Spectrophotometry.

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INTRODUCTION

Analytical chemistry may be defined as the science and art of determining the composition of materials in terms of elements of compounds contained[1]. Analysis is important in every product or service but in, drug analysis is important as it involves life. Quality of product comes from series of tests from quality control starting from raw material, in process during finished product etc[2]. Development of the simple and reproducible analytical methods for estimation of drugs is very important part of quality control and for social awareness which is established in present work[3].

Fluoxetine[4] has activating properties that make it a good option for patients with retarded depression or atypical depression. As other selective serotonin reuptake inhibitors, fluoxetine inhibits the serotonin transporter protein. In addition, it is also a weak norepinephrine reuptake inhibitor, this effect increases with higher dose. Fluoxetine is an antagonist of $5HT_{2C}$ receptors; this has been proposed as potential mechanism for its activating properties.

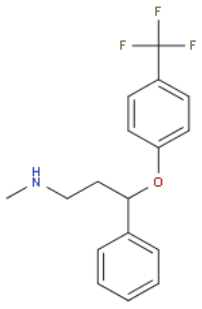

Figure 1: Fluoxetine

MATERIALS AND METHODS

Chemicals and Reagents: Fluoxetine was kindly gifted by Sun Pharmaceuticals Pvt Ltd, Hyderabad certified to contain 99.9% purity respectively. The drug was used without further purification. All the

solvents used in analysis were of Spectroscopic grade.

Prodep 20 capsules (label claim 20 mg of Fluoxetine) of Sun Pharma Laboratories was used in analysis.

UV- spectrophotometry: UV- spectrophotometer (SYSTRONICS-2203) with spectral bandwidth of 2 nm and 10 mm matched quartz cells was used.

Preparation of standard stocks solution: Standard stock solution of fluoxetine was prepared by dissolving 50 mg of drug in 50 ml of water and diluted to required volume with same solvent. Then the solution was further diluted to get concentration of 10µg/ml. The stock solution was suitably diluted with water to get a concentration range from 5-35µg/ml and their absorbance was measured at 224nm. Using the absorbance values against concentration calibration curve was plotted. From the graph it was found that fluoxetine obeys beers law between 5-35µg/ml.

Analysis of Formulation: The powdered drug from 10 capsules was taken. An accurately weighed quantity of capsule powder equivalent to 50 mg of fluoxetine was transferred to 50 ml standard flask. The content of flask mixed with 30 ml of water and shaken to dissolve the active ingredients and then made up to the volume with water. The solution was filtered and then filtrate diluted with water to give the concentration range 20µg/ml. absorbance values of sample solution was measured at 224nm.

Formula used for calculation:

$$\text{Average weight} = \frac{\text{total wt of powder in capsules}}{\text{total no. of capsules}}$$

Amount present =

$$\frac{\text{sample absorbance}}{\text{standard absorbance}} \times \frac{50}{\text{wt of sample}} \times \frac{50}{1} \times \frac{\text{wt of standard taken}}{50} \times \frac{1}{50} \times \text{average weight}$$

RESULTS AND DISCUSSION

Determination of λ max: Standard stock solution of fluoxetine was prepared by dissolving 50 mg of drug in 50 ml of water and diluted to required volume with same solvent. Then the solution was further diluted to get concentration of 10µg/ml. The solution was scanned in UV region from 200-400 nm. The λ max of drug was found to be at 224 nm

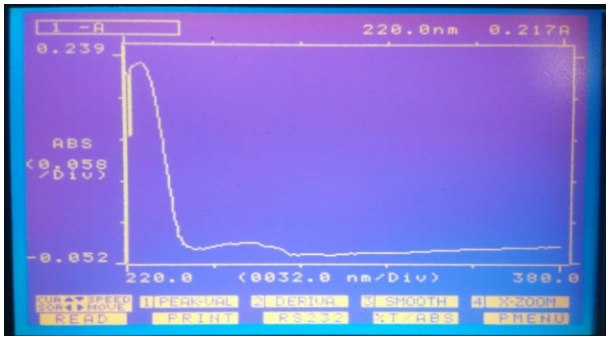

Figure 2: spectra of standard

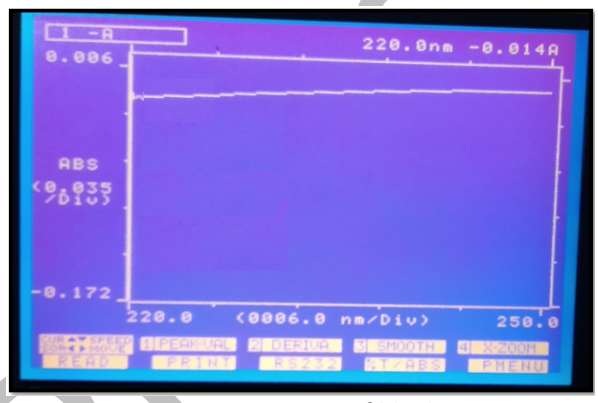

Figure 3: spectra of blank

Table 1: Determination of λ max

S.no	Wavelength (nm)	Absorbance (abs)
1	220	0.295
2	221	0.316
3	222	0.342
4	223	0.375
5	224	0.397
6	225	0.362
7	226	0.344
8	227	0.316
9	228	0.296
10	290	0.290

Validation Parameters

Linearity: Using the absorbance values against concentration calibration curve was plotted. From the graph it was found that fluoxetine obeys beers law between 5-35µg/ml.

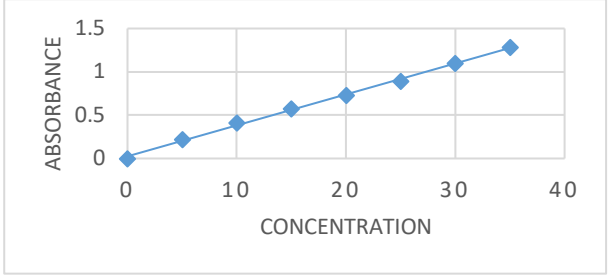

Figure 4: Calibration Graph of Fluoxetine

Table 2: Linearity Values of Fluoxetine

S.no	Concentration (µg/ml)	Absorbance (abs)
1	5	0.216

Table 3: Quantitative Estimation Values of Fluoxetine

S.no	Concentration µg/ml	Label claim	Amount present	% of label claim	% deviation
1	10	20	0.02030	101.1%	-0.011
2	15	20	0.01965	98.4%	0.016
3	20	20	0.02008	100.5%	-0.005
4	25	20	0.0203	101.6%	-0.016
5	30	20	0.0196	99%	0.01

2	10	0.409
3	15	0.577
4	20	0.725
5	25	0.892
6	30	1.099
	Slope	**0.035**
	Correlation coefficient	**0.998**

Precision: The closeness of agreement between a series of measurements. Multiple sampling of homogenous samples under prescribed condition, precision is of two types which includes Repeatability and Reproducibility.

Table 4: System Precision

S.no	Repeatability (conc) µg/ml	Absorbance (abs)
1	20	0.739
2	20	0.755
3	20	0.761
4	20	0.768
5	20	0.761
6	20	0.765
Average		0.758167
Standard deviation		0.010362
%RSD		0.013667

Table 5: Method Precision

S.NO	CONC (µg/ml)	ABS	AVG
1	20	0.739	0.7355
2	20	0.736	0.7375
		0.739	
3	20	0.776	0.775
		0.774	
4	20	0.768	0.768
		0.768	
5	20	0.734	0.733
		0.732	
6	20	0.729	0.7305
		0.734	
Average			**0.74675**
Standard deviation			**0.01948**
%RSD			**0.02599**

Repeatability (system precision): 20µg/ml concentration solution of fluoxetine was prepared whose absorbance was measured six times for which relative standard deviation is calculated. The relative standard deviation for preparation should not be more than 2%.

Reproducibility (method precision): Six individual preparations of fluoxetine was prepared with a concentration of 20µg/ml. Whose absorbance measured at 224nm. The relative standard deviation for preparation should not be more than 2%.

Accuracy: The sample solution was prepared to get a concentrate range of 15µg/ml, 20µg/ml, 25µg/ml, into which 5 mg of pure powder has been added to get 90%, 100%, 110% concentration range. The percentage recovery is calculated for these concentrations from absorbance obtained. The percentage criteria for the spiked preparation should be within 98-102%.

The percentage recovery was calculated by using the following formula;

$$\text{Percentage recovery} = \frac{amount\ obtained}{amount\ added} \times 100$$

Table 6: Accuracy Values of Fluoxetine

S.no	Conc	Mg added	Mg found	% recovery
1	90	5	4.950	99.10%
2	100	5	5.10	101.90%
3	110	5	5.009	100.186%

Limit of Detection: The detection limit of an individual analytical procedure is the lowest amount of analyte in a sample which can be detected but not necessarily quantified as an exact value.

Table 7: LOD of Fluoxetine

S.NO	PARAMETER	FLUOXETINE
1	Slope	0.0351
2	Standard deviation	0.01948
3	LOD	1.8366

Limit of quantitation: The limit quantitation of an individual analytical procedure is the lowest amount of analyte in a sample which can be quantitatively determined with precision and accuracy

LOQ = 10 × S.D/SLOPE

= 10 × 0.01948/0.0351= 5.56µg/ml

Table 8: LOQ of Fluoxetine

S.NO	PARAMETER	FLUOXETINE
1	Slope	0.0351
2	Standard deviation	0.01948
3	LOQ	5.56

Solution Stability: 20µg/ml concentration solution of fluoxetine was prepared and the solution whose absorbance was measured for every half an hour for 90 minutes and the solution was found to be stable up to 90 min.

Table 9: Stability Values of Fluoxetine

S.NO	TIME (min)	ABSORBANCE
1	0	0.746
2	30	0.731
3	60	0.729
4	90	0.720

CONCLUSION

On the basis of our experimental results, we concluded that the UV spectrophotometric method developed for the determination of fluoxetine is found to be precise, accurate and cost effective. Hence this method can be used for routine analysis of fluoxetine in bulk and pharmaceutical dosage form.

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