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ANALYTICAL METHOD DEVELOPMENT AND VALIDATION OF ESCITALOPRAM OXALATE, USING UV-VISIBLE SPECTROSCOPY: SINGLE POINT STANDARDIZATION, STANDARD ABSORPTIVITY, AND CALIBRATION CURVE APPROACH

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ABSTRACT

UV spectrophotometric techniques play a crucial role in pharmaceutical analysis for the estimation of active compounds. This study presents the development and validation of a UV-Visible Spectroscopy method for escitalopram oxalate using methanol as a solvent. The method based on Beer-Lambert's law and ICH guidelines, show a maximum absorbance at 243 nm. Excellent linearity (R^2 = 0.997) was observed over the tested concentration range, with precise LOD and LOQ values confirming the method's sensitivity. Additionally, the method demonstrated high precision, accuracy, and robustness, making it a reliable choice for routine pharmaceutical quality control applications.

KEYWORDS: Escitalopram oxalate, UV spectroscopy, Validation, Accuracy, Precision.

INTRODUCTION[1-23]

Escitalopram Oxalate (ESC) is a well-known selective serotonin reuptake inhibitor (SSRI) used to treat anxiety, depression, and other related conditions.[1-3] Chemically, ESC is (1S)-1-(3-(dimethylamino) propyl)-1-(4-fluorophenyl)-3H-2-benzofuran-5-carbonitrile, combined with oxalic acid (Figure 1).

Several analytical techniques have been documented in the literature for the measurement of ESC, both alone and in combination with other compounds in pharmaceutical preparations. The methods include ultraviolet spectrophotometry (UV),[4-9] reverse-phase high-performance liquid chromatography (RP-HPLC),[10-19] and thin-layer chromatography (TLC) with high performance.[20-22]

However, the previously reported methods are often time-consuming and costly. Therefore, developing a simple, precise, rapid, and cost-effective method for the quantification of ESC in tablet dosage forms is considered highly valuable.[23]

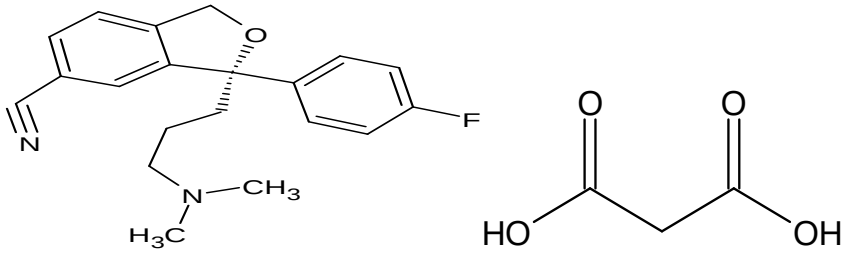

Fig. No. 1: ESC.

MATERIAL AND METHOD

Instrumentation

A Systronics (India) Limited UV-visible spectrophotometer, Model AU-2707, with a 1 cm cuvette and a double beam configuration system was used for the study. An ultrasonicator cleaner was used to degas the solvent. For the weighing, an electronic balance was used.

Chemicals or reagents

Sydler Remedies Pvt. Limited provided an analytically pure sample of ESC, Nexito 10 a tablet formulation produced by Sun Pharmaceutical Industries Ltd, was purchased from a nearby pharmacy.

METHODOLOGY

Selection of suitable solvent

The drug solubility, stability, and absorbance maxima of the chemical in the specific solvent were taken into consideration when choosing the solvent. ESC (10 mg) was weighed, and its solubility was examined in distilled water, 0.1N hydrochloric acid, 0.01N sodium hydroxide, methanol, ethanol, and phosphate buffer pH 6.8. Methanol is the more effective solvent from the list above. Methanol is therefore utilized as a solvent.

Preparation of standard stock solution

After being weighed, 50 mg of pure ESC was moved to a 50 ml volumetric flask and dissolved in methanol. To get a final concentration of 1000 ppm, it was thoroughly dissolved and diluted with diluent. A 10 ppm solution was made from the stock solution using methanol, which served as the working standard.

Determination of wavelength

ESC was synthesized at a concentration of 10 ppm, and its UV spectra were scanned between 200 and 400 nm to determine the wavelength maxima. This wavelength was selected for the investigation. It was found that the maximum absorbance against methanol is 243 nm.

Estimation of ESC Tablet

In a glass mortar, twenty ESC tablets were weighed and grind into powder. After transferring the amount of powder equal to 10 mg of ESC Tablet into a 10 ml volumetric flask, it was dissolved in around 5–6 ml of methanol and placed in an ultrasonicator for 15 minutes. The flask was then diluted with methanol to reach a concentration of 1000 ppm. Whatmann filter paper was used to filter the mixture. As previously mentioned, the sample solution was diluted and examined.

Table No. 1: Drug estimation.

Coc^n (API)	**Abs (API)**	**Coc^n (Tablet)**	**Abs at 243 nm**	**Specifications**
4 ppm	0.292	10 ppm	0.687	**Brand name:** NEXITO 10
8 ppm	0.566	10ppm	0.689	**Label claim:** 10mg
12 ppm	0.789	10 ppm	0.681	**Mfg by:** Sun Pharmaceutical Industries Ltd,
16 ppm	1.021	Mean	0.6856	**Manufacturing date:** Feb 2024
20 ppm	1.269	SD	0.0041	**Expiry date:** Feb 2029
		RSD	0 .0059	**Batch no:** EMS1887
		%RSD	0 .59%	**% Purity of drug:** 95.8 %.

Estimation of ESC by using Standard Absorptivity Method

Prepare standard solutions of ESC in methanol at concentrations ranging from 4 ppm to 20 ppm. Measure the absorbance of each solution at 243 nm using a UV-visible spectrophotometer. Calculate the absorptivity (a) using Beer-Lambert's law

A = abc

Where: ***A*** *= Absorbance,* ***a*** *= Molar Absorptivity,* ***b*** *= Path length (1 cm),* ***c*** *= Coc^n in ppm*

The standard absorptivity method is based on the principle of Beer-Lambert's law, which states that absorbance is directly proportional to concentration by determining the absorptivity coefficient (a), unknown sample concentrations can be estimated accurately. This method is widely used due to its simplicity and ability to provide reliable quantitative results. By using formula it was noted that the percentage purity of drug by using this method was found 95.8%. (By using Table No. 1)

Estimation of ESC by using single point method[24]

Prepare a standard solution of ESC in methanol at a known concentration (e.g., 10 ppm). Prepare the test sample solution in methanol. Measure the absorbance of both the standard and test sample at 243 nm using a UV-visible spectrophotometer. Calculate the concentration of the test sample using the equation:

Ctest = (Atest X Cstd) / Astd

Where, Ctest = concentrations of sample
Cstd = concentrations of standard
Atest = absorbance of the sample
Astd = absorbance of the standard

The Single Point Standardization Method is a direct and simple approach for estimating the concentration of an unknown sample by comparing its absorbance with that of a standard solution of known concentration. It is particularly useful for quick and routine analysis where high precision is not required. By using formula it was noted that the percentage purity of drug by using this method was found 95.8%
(By using Table No. 1)

Calibration curve method[24]

Prepare standard solutions of ESC in methanol at concentrations ranging from 4 ppm to 20 ppm. Measure the absorbance of each solution at 243 nm using a UV-visible spectrophotometer. Plot a calibration curve by graphing absorbance against concentration. Determine the equation of the calibration curve using linear regression analysis. Use the calibration curve equation to estimate the concentration of unknown samples by interpolating their absorbance values.

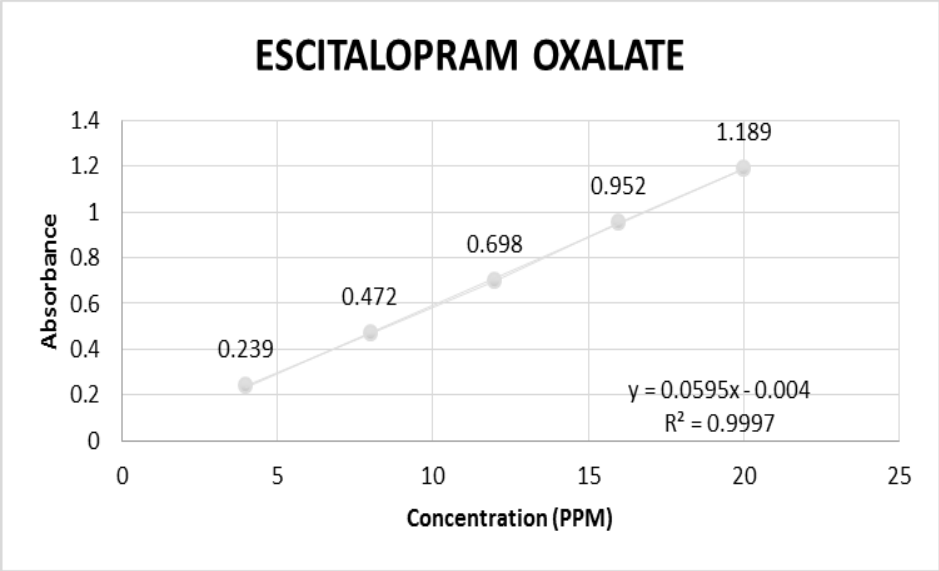

Fig. No. 2: Calibration Curve ESC.

The Calibration Curve Method is based on Beer-Lambert's law, where absorbance is directly proportional to concentration. By constructing a standard calibration curve, the concentration of unknown samples can be accurately determined. This method ensures high precision and reliability in quantitative analysis. By using equation of line after calculating by using formula it was noted that the percentage purity of drug by using this method was found 95.8 %.

Method of validation[25-38]

Linearity[25-27]

Using methanol as a blank, the absorbance of each concentration was determined at 243 nm. Standard stock-2 solution with concentrations ranging from 4 to 20 ppm was used to create new aliquots. The linearity curve's regression coefficient (R2) value was determined to be 0.9997. The results of linearity are shown in Table 1.

Table No. 02: Linearity.

Concentration	Absorbance	Calculation	
4	0.239	MEAN	0.71
8	0.472	MODE	N/A
12	0.698	MEDIAN	0.698
16	0.952	SD	0.3763
20	1.189	RSD	0.53
TABLET – 10 PPM	0.687	%RSD	53 %
		C.C	0.9997
		INTERSECT	(- 0.004)
		SLOPE	0.0595

Precision[28-29]

The degree of repeatability of an analytical method under normal operating conditions. It is divided into:

Intraday and Interday precision

Results showed minimal variation in absorbance values over different time intervals and on different days, demonstrating the method's precision.

Table No. 03: Intra day precision.

Time	Concentration	Absorbance	Calculation
0 Hrs.	12PPM	0.698	Mean - 0.6993
2 Hrs.	12PPM	0.702	SD – 0.0047
4 Hrs.	12PPM	0.691	RSD – 0.00682
8 Hrs.	12PPM	0.705	%RSD- 68%
12 Hrs.	12PPM	0.699	
24 Hrs.	12PPM	0.701	

Table No. 04: Inter day precision.

Time	Concentration	Day 1	Day 2	Day 3
0 Hrs.	12PPM	0.698	0.691	0.701
2 Hrs.	12PPM	0.702	0.700	0.706
4 Hrs.	12PPM	0.691	0.688	0.683
8 Hrs.	12PPM	0.705	0.701	0.687
12 Hrs.	12PPM	0.699	0.689	0.681
24 Hrs.	12PPM	0.701	0.685	0.680
	Mean	0.6993	0.6923	0.6896
	SD	0.0047	0.0066	0.0110
	RSD	0.0068	0.0095	0.0161
	%RSD	0.68%	0.95%	1.61

Accuracy[30-31]

Recovery studies at various replicate levels in triplets for 80%, 100%, and 120% were used to test the accuracy of the suggested method. The sample solutions were made by adding a known quantity of pure drug to the pre-analyzed formulation, and the mean percent recovery was calculated and reported in ESC.

Table No. 5: Accuracy.

Spiked Level (%)	Added Concentration	Measured Concentration	% Recovery
80	8 ppm	7.9	98.8
100	10 ppm	10.1	101
120	12 ppm	12.2	101.7

Result shows mean of 3 readings

Ruggedness[32-34]

Two different analysts used to perform the analysis, and the absorbance of each was recorded in order to find out the % RSD. Results are provided in Table no 06:

Table No. 6: Ruggedness.

Analyst – 1		Analyst – 2	
Concentration (PPM)	**Absorbance**	**Concentration (PPM)**	**Absorbance**
12 PPM	0.690	12 PPM	0.695
12 PPM	0.680	12 PPM	0.691
12 PPM	0.689	12 PPM	0.681
12 PPM	0.675	12 PPM	0.675
12 PPM	0.670	12 PPM	0.677
12 PPM	0.682	12 PPM	0.679
Average	0.681	Average	0.683
SD	0.0077	SD	0.0080
RSD	0.0113	RSD	0.0111
% RSD	1.1	% RSD	1.1

Robustness[35-36]

Three separate wavelengths of analysis were used to determine the method's robustness. The relative absorbance was noted, and the findings were shown in Table no. 07.

Table no. 07: Robustness.

Concentration (PPM)	Wavelength (nm)		
12	241	243	245
12	0.694	0.698	0.701
12	0.696	0.699	0.702
12	0.701	0.703	0.706
12	0.707	0.708	0.713
12	0.711	0.712	0.715
AVERAGE	0.702	0.704	0.703
SD	0.0064	0.0053	0.0057
%RSD	0.64%	0.53%	0.57%

Limit of Detection (LOD) and Limit of Quantification (LOQ)[37-38]

LOD is the lowest amount of analyte that can be detected but not necessarily quantified under stated conditions and LOQ is the lowest amount of analyte that can be quantitatively determined with acceptable precision and accuracy.

$LOD = \frac{3.3\ X\ \sigma}{S}$ $LOQ = \frac{10\ X\ \sigma}{S}$

Where, σ = Standard deviation of the response

S = Slope of the calibration curve

LOD and LOQ were calculated as 0.098 PPM and 0.297 PPM, respectively.

DISCUSSION

Escitalopram oxalate was scanned in the UV region between 200 and 400 nm with methanol as the solvent in order to develop the UV-spectrophotometric method. The maximum wavelength was recorded at 243 nm. This approach has been validated in accordance with ICH criteria. Escitalopram oxalate is analysed and determined using a variety of characteristics, including linearity, accuracy, precision, robustness, ruggedness, LOD, and LOQ. For linearity investigations, various doses between 4 and 20 µg/ml were generated from the same solution, and the linearity curve's R2 value was 0.9997. was found that the mean percentage recovery fell within the range that indicated the correctness of the method's development. the accuracy parameter was evaluated at 80%, 100%, It and 120%. The linearity curve was used to calculate the parameters LOD and LOQ.

RESULT AND CONCLUSION

A straightforward UV-spectrophotometric technique for ESC has been created and verified in accordance with ICH guidelines. Our investigation's results demonstrated that the suggested UV-spectrophotometric approach was incredibly sensitive, accurate, and reasonably priced when compared to the previously published techniques. The suggested UV-spectrophotometric method proved to be practical for determining escitalopram oxalate in bulk.

Conflict of interest

Regarding this article, the authors disclose no relevant conflicts of interest.

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