



## Stability Indicating Spectrofluorimetric Method for the Estimation of Escitalopram in Bulk and Pharmaceutical Dosage Form

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### ABSTRACT

**Aim:** The purpose of this research was to design and validate a reliable, sensitive, and accurate spectrofluorimetric approach for the determination of Escitalopram. **Materials and Methods:** Acetonitrile was used as the solvent, and the  $\lambda_{\text{max}}$  was determined to be 239 nm. The technique has been applicable for accuracy, limit of detection (LOD), linearity, limit of quantification (LOQ), as well as precision. **Results:** On the basis of the results, escitalopram had a correlation coefficient of 0.9992 as well as was linear in 1–9  $\mu\text{g/mL}$  range. Value of LOD had been 0.112  $\mu\text{g/mL}$ , whereas LOQ had been 0.370  $\mu\text{g/mL}$ . Stability studies demonstrated that Escitalopram was vulnerable to alkaline, acid, along with oxidative degradation. **Conclusion:** The developed spectrofluorimetric method has been discovered to be sensitive, simple, and appropriate for Escitalopram quantification. The method met the ICH guidelines for linearity, precision, LOD, accuracy, and LOQ, making it suitable for regular quality inspection of Active Pharmaceutical Ingredient (API) and formulations.

**Keywords:** Acetonitrile, Antidepressant, Escitalopram, Methanol, Spectrofluorimetry.

### INTRODUCTION

It is an antidepressant medication primarily used to treat mental health conditions. It works by increasing serotonin levels, a chemical messenger that regulates mood, in the brain. By restoring balance to the brain's chemical messaging system, escitalopram helps alleviate symptoms of depression and anxiety.<sup>1,2</sup> Citalopram's S enantiomer is escitalopram as well as more potent than citalopram's R-enantiomer.<sup>3</sup> Escitalopram has been chemically (1S)-1-[3-(dimethylamino) propyl]-

1-(4-fluorophenyl)-3H-2-benzofuran-5-carbonitrile with having 324.4 g/mol molecular weight and it has been in the form of solid which appears as fine white to slightly-yellow in colour and is shown in Fig. 1. It has the melting point of 147-155°C and its Pka value is 9.78.<sup>4</sup> Marketed formulations are Cipralex and Lexapro.<sup>5</sup> Escitalopram Oxalate (ESC) is a widely used antidepressant medication, and its accurate determination in pharmaceutical formulations is crucial for ensuring its efficacy and safety. There have been reports of various analytical approaches for ESC determination, including spectrophotometric



methods,<sup>6-8</sup> chromatographic methods,<sup>9-14</sup> as well as electrochemical methods.<sup>15</sup> Additionally, for simultaneous quantification of ESC along with its metabolites, UHPLC-MS/MS techniques have been established.<sup>16</sup> However, many of these methods have restrictions, for example, narrow linear ranges, low sensitivity, and the requirement of expensive instruments. As a consequence, a straightforward, accurate, and sensitive analytical technique for determining ESC in pharmaceutical formulations must be developed.

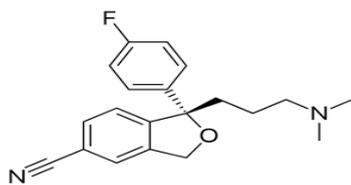


Fig. 1. Chemical Structure of Escitalopram  
**MATERIALS AND METHODS**

## Experimental procedure

### Materials

Escitalopram, Water, Methanol, Dimethyl sulfoxide, Dimethyl formamide, Acetonitrile.

### Instruments

Analytical Balance, Melting point apparatus, FTIR, UV Spectrophotometer, Spectrofluorophotometer.

### Spectrofluorimetric method

#### Solvent selection

During the initial stages of method development for ESC, various solvents were examined, including water, methanol, acetonitrile, DMSO, and DMF. In order to determine the optimal solvent for determining escitalopram, a variety of solvents were used for the solubility experiments. It was shown that escitalopram was readily soluble in methanol and acetonitrile, among other substances. Acetonitrile is utilized for all of the dilutions in this experiment. Because of its improved solubility and reproducible maximum absorbance readings, acetonitrile was considered for additional study.

### Development and validation of analytical method

#### Preparation of Escitalopram standard solution

Escitalopram's stock solution (100 µg/mL) had been made by dissolving 10 mg of drug in a 10 mL volumetric flask, then diluting to volume with acetonitrile. To achieve the desired concentration, 1

mL of this stock solution had been transferred to a second 10 mL volumetric flask as well as diluted to volume with acetonitrile.

### Wavelength Selection

Calculated excitation wavelength in normal solution is ( $\lambda_{exc}$ ) 281 nm. The emission spectra were obtained using this excitation intensity. It has been estimated that the emission wavelength is ( $\lambda_{emi}$ ) 298 nm. This led to the measurement of Escitalopram fluorescence intensity at these wavelengths.

### Method validation

ICH guidelines had been followed in the validation of developed approach, demonstrating satisfactory linearity, accuracy, precision, and LOD and LOQ. This validation ensures the method's flexibility for various concentrations and formulations, eliminating the need for re-validation. The validation process assessed the method's suitability for future applications.

### Linearity

Linearity of fluorescence response had been assessed through plotting concentration against corresponding fluorescence intensity. A series of standard solutions (10 µg/mL) had been made through dilution of the stock solutions. Calibration curves were constructed by graphing fluorescence intensity versus concentration, yielding regression equations.

### Escitalopram standard solution Preparation

Standard stock solution of escitalopram (100 µg/mL) had been made through dissolving 10 mg in 10 mL acetonitrile, then further diluted to concentrations ranging from 1-9 µg/mL for Spectrofluorimetric analysis.

### Accuracy

By using the conventional additive approach to calculate ESC restoration, the strategy's validity has been evaluated. 80%, 100%, and 120% of the standard Escitalopram solution have been added to the powdered form of the medication. By examining the fluorescence spectra at  $\lambda_{EX}$  at 281 nm and  $\lambda_{EM}$  at 298 nm, one can estimate the value of ESC. The recovery process was verified by calculating the medication three times at each designated dosage level.

### Precision

Intra-day precision of proposed Spectro-

fluorimetric technique had been evaluated through analysing three concentrations of escitalopram (1, 5, and 9 µg/mL) with multiple replicates on the same day. Comparable ESC levels have also been used to evaluate the inter-day precision of the suggested Spectrofluorimetric technique, and the associated response was measured on alternative days of the week. The precision results within and between days were presented as a percentage of standard deviation (%RSD).

#### Limit of Detection and Limit of Quantification

A sample with extremely low analyte concentrations was used to test the procedure's LOD and LOQ in compliance with the International Conference on Harmonization.

The LOD and LOQ were established by following equations

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

Where,

$\sigma$  - blank's standard deviation and  
S - Calibration plot's slope.

#### Assay

The average weight of five Escitalopram tablets was determined, and subsequently, the tablets were pulverized into fine powder using a mortar and pestle. 10 milligrams of tablet powder were taken in 10 millilitre volumetric flask as well as dissolved in acetonitrile. One millilitre of the stock solution mentioned above is diluted with ten millilitres of solvent after the solution has been filtered with What-man filter paper. Two strengths of 3 µg/mL and 5 µg/mL solution are made from these solutions. By measuring the intensity at the emission wavelength at 298 nm the amount of escitalopram is determined. The assay percentage needs to be between 98.0% and 102.0%.

#### Stress testing studies

In order to do stress testing investigations, the sample was subjected to an acidic, oxidative, heat, UV, alkaline environment.

#### Acid degradation

Weigh 10 mg escitalopram and add to 10 mL acetonitrile in 10 mL volumetric flask. 10 mL of 1N HCl is added to 1 mL of stock solution in a different container. Pour 1mL of intermediate solution into a 10 mL volumetric flask, then add 1N NaOH and utilize acetonitrile to dilute it to

volume. Make highest concentration range and scan spectrofluorimetrically after 1 hour.

#### Alkaline degradation

Transfer 10 mg of escitalopram to a 10 mL volumetric flask after weighing it. Make a 1000 µg/mL stock solution by adding 10 mL of acetonitrile. In a different 10 mL volumetric flask, move 1 mL of the stock solution as well as add 10 mL of 1N NaOH. From them, 1 millilitre of solution is taken, 1 millilitre of 1N HCl is added, and the volume is adjusted using acetonitrile. Now, one of the linearity concentration ranges was created by diluting the necessary amount and was scanned in the device after 1 hour.

#### Oxidative degradation

Weigh 10 mg of escitalopram as well as transfer it to a 10 mL volumetric flask. Utilize a solution of 3% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) to dissolve along with dilute to volume. Transfer 1mL of escitalopram stock solution to 10 mL volumetric flask. Utilizing 9 mL of acetonitrile to dilute to volume. A suitable dilution was made to create one of the linearity concentration ranges, which was then scanned in a spectrofluorimeter every hour.

#### Thermal effect

After being precisely weighed, 30 mg of the medication is heated to 105°C for 24 h in the hot air oven. The sample stock solution is made from the aforementioned medication using the proper dilutions (100 µg/mL). By generating the proper dilution, one of the concentration ranges of linearity was now generated and scanned in a spectrofluorimeter.

#### UV effect

30 mg of the medication was precisely weighed, and it was stored for 24 h in a UV cabinet at a fixed short wavelength (254 nm). Ten milligrams of the aforementioned medication are combined with ten millilitres of acetonitrile. Now, by making the proper dilution and scanning in a spectrofluorometer, one of the highest concentration ranges of linearity (9 µg/mL) was generated.

## RESULTS AND DISCUSSION

#### Drug authentication studies

##### Determination of IR spectra

The FTIR Spectra of Escitalopram were obtained, which are shown in Fig. 2. The FTIR Spectral bands of Escitalopram were compared with the reference values and publicized in Table 1.

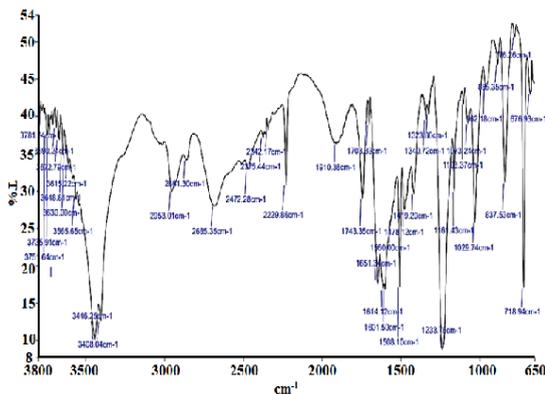


Fig. 2. IR Spectra of Escitalopram

Table 1: Characteristic bands of Escitalopram

Sr.No	Functional group	IR bands (cm <sup>-1</sup> )	
		Measured value	Standard value
1	C≡N	2229	2250-2100
2	C-O-C	1233	1250-1050
3	C-F	1029	1400-1000
4	C=C	1651	1680-1640
5	C-C	837	1300-800

**Choice of Solvent**

The solubility data of Escitalopram is presented in Table 2.

Table 2: Solubility data

Solvent	Solubility
Water	Insoluble
Methanol	Soluble
Acetonitrile	Soluble
DMSO	Soluble

**Determination of UV ( $\lambda_{max}$ )**

UV absorbance spectrum of Escitalopram was obtained in the  $\lambda$  range of 200-400 nm shown in Fig. 3, and the  $\lambda_{max}$  for Escitalopram was found to be 217 nm.

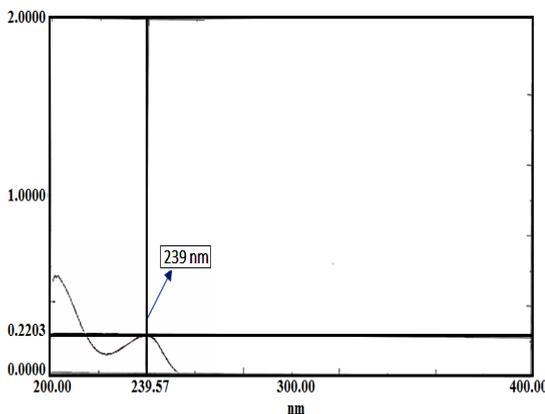


Fig. 3. UV Spectrum of Escitalopram

**Selection of Emission wavelength**

In a 1 cm cell, the solution was scanned between 220 and 770 nm. It was found that the emission wavelength was 298 nm and the excitation wavelength was 281 nm showed in Figure 4.

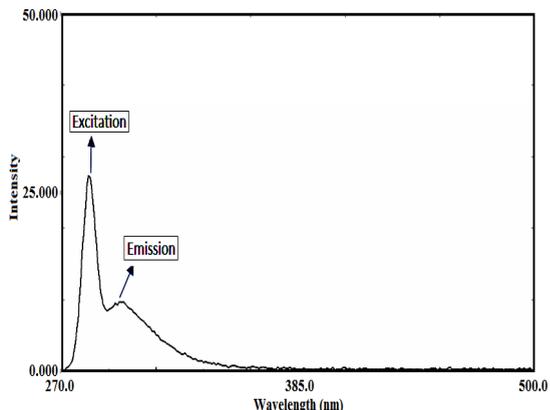


Fig. 4. Excitation and Emission Wavelength

**Method validation**

**Linearity**

Concentrations ranging from 1 to 9 micrograms per millilitre shown in Table 3 were used to test linearity. The emission spectra of Escitalopram are shown in Fig. 5. It was discovered that the Y value was  $1.6232x + 0.558$  which was illustrated in Figure 6.

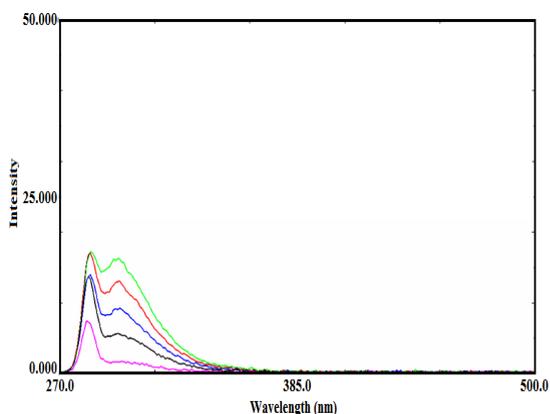


Fig. 5. Emission spectra of Escitalopram

Table 3: Linearity data

Sr. No	Concentration	Fluorescence Intensity
1	1	2.003
2	3	5.655
3	5	8.715
4	7	11.868
5	9	15.128

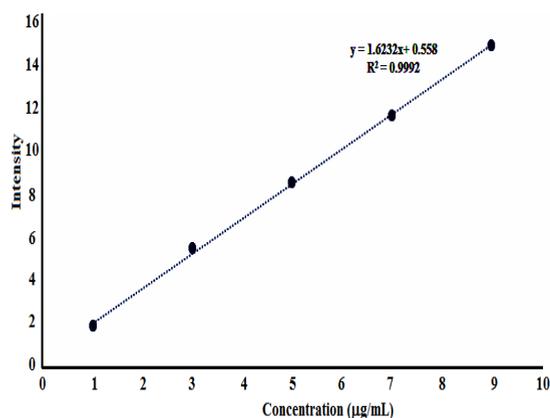


Fig. 6. Calibration plot

### Accuracy

Various spiked concentrations were used to test accuracy. Spiked values of 80%, 100%, and 120% were measured. The Escitalopram recovery rate ranged from 100.9-102.5%, which

Table 4: Accuracy Data of Escitalopram

Spiked level	Conc of the sample (µg/mL)	Conc of the standard (µg/mL)	Total Conc (µg/mL)	Amt recovered (µg/mL) AM±SD (n=3)	Recovery(%)	%RSD
80%	1	0.8	1.8	1.839±0.032	102.2	1.740
100%	1	2	2	2.050±0.040	102.5	1.951
120%	1	1.2	2.2	2.219±0.025	100.9	1.126

Table 5: Precision data for Escitalopram

Concentration Milligram/millilitre	Intra-day		Inter-day	
	Conc (µg/mL) (AM±SD)	%RSD	Conc (µg/mL) (AM±SD)	%RSD
1	2.132±0.008	0.375	2.334±0.042	1.799
5	8.314±0.090	1.082	8.197±0.096	1.171
9	15.44±0.129	0.835	15.144±0.049	0.323

Table 6: LOQ and LOD data

LOD (µg/mL)	112
LOQ (µg/mL)	370

Table 7: Assay data of Escitalopram

Drug	Brand	Label Claim	Amount found ±SD (n=3)	Assay%	%RSD
Escitalopram	NEXITO	10 mg	9.92±0.208	99.2	0.209

### Stress testing studies

#### Acid degradation

The acid degradation study revealed that Escitalopram underwent significant degradation, with a 29% reduction in intensity after Exposure to 1N HCl indicating its susceptibility to acidic conditions, and the spectra of acid degradation are shown in Figure 7.

#### Alkaline degradation

The alkaline stress testing shows

is listed in Table 4.

### Precision

Three distinct levels of Escitalopram solutions of different strength (1, 5, 9 microgram/millilitre) were analysed intra-day (n=3) to assess the method's repeatability and the above concentrations were analysed over the course of three days, and the %RSD for Escitalopram is lower than 2 which was shown in Table 5. Statistical data showed high degree of precision.

### Limit of Quantification and Limit of Detection

The LOD and LOQ values are calculated, and the values are listed in Table 6.

### Assay

The assay of the drug was conducted, and the results obtained were within the acceptable limits presented in Table 7.

Escitalopram is prone to alkaline-catalysed degradation, with a loss of 17.7% of its initial intensity after treating it with 1N NaOH as shown in Fig. 8, underscoring the need for careful consideration of alkaline conditions in pharmaceutical applications.

### Oxidative degradation

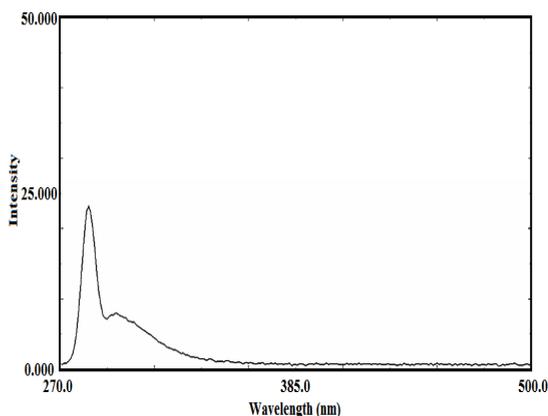
The oxidation degradation study using 3% H<sub>2</sub>O<sub>2</sub> resulted in 35.6% degradation of Escitalopram, as evidenced by reduced intensity which was represented in Fig. 9, highlighting the importance of antioxidant considerations in formulation and storage.

### Thermal effect

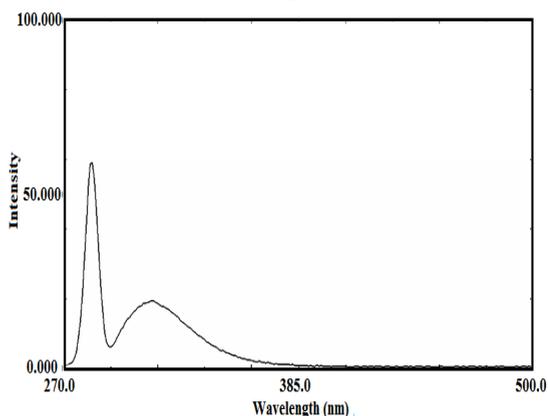
Escitalopram demonstrated remarkable thermal stability, exhibiting no discernible degradation after 24 h exposure to elevated temperatures in a hot air oven. The thermal spectra of Escitalopram is shown in Figure 10.

**UV Effect**

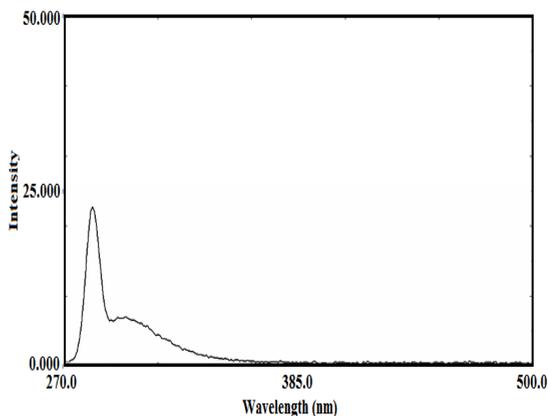
The photostability study showed that Escitalopram was resistant to UV-induced degradation, with no degradation products formed after 24 h of exposure to UV radiation as shown in Figure 11.



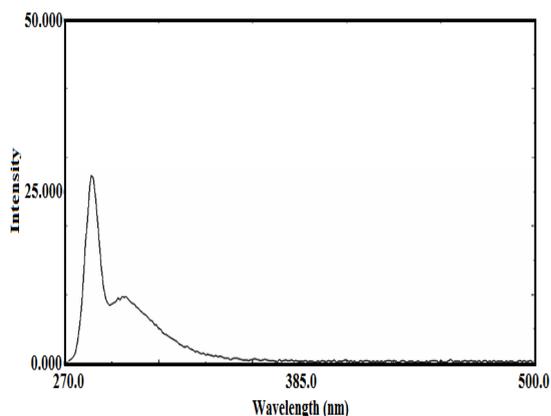
**Fig. 7. Acid degradation spectra of Escitalopram**  
Alkaline degradation



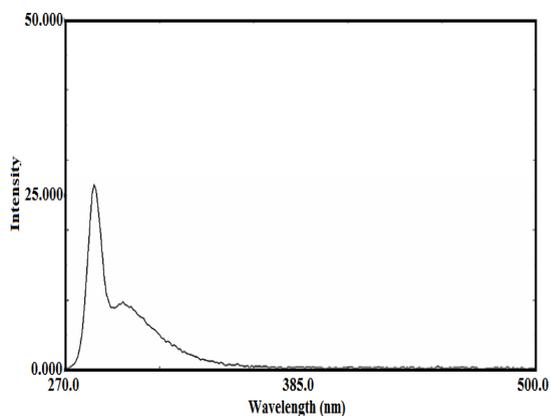
**Fig. 8. Alkaline degradation spectra of Escitalopram**  
Oxidative degradation



**Fig. 9. Oxidative degradation spectra of Escitalopram**



**Fig. 10. Thermal Effect spectra of Escitalopram**



**Fig. 11. UV Effect spectra of Escitalopram**  
**CONCLUSION**

A novel, simple and sensitive Spectrofluorimetric method was developed for estimating escitalopram in tablet and bulk dosage forms. The method exhibited linearity over 1-9  $\mu\text{g/mL}$  at an emission wavelength of 298 nm. Calibration plot analysis demonstrated accuracy through commercial formulation analysis, yielding percentage recovery within acceptable limits. Intra-day, as well as inter-day precision assays, showed  $\%RSD < 2$ , indicating good precision. LOD and LOQ were 0.112  $\mu\text{g/mL}$  along with 0.370  $\mu\text{g/mL}$ , correspondingly. Assay percentage was calculated as 93.2%. Degradation studies revealed that escitalopram underwent significant degradation under acidic (29%), alkaline (17.7%), and oxidative (35.6%) stress conditions while remaining stable under thermal and UV exposure. Compared to chromatography, this Spectrofluorimetric method offers superior clarity, precision, and accurate results. Its simplicity, effectiveness, and minimal equipment requirements make it an attractive alternative.

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**Conflicts of interest**

The authors declare that there is no conflict of interest.

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