IJPPR
INTERNATIONAL JOURNAL OF PHARMACY & PHARMACEUTICAL RESEARCH
An official Publication of Human Journals
ISSN 2349-7203
HUMAN

Human Journals
**Research Article**
November 2016 Vol.:7, Issue:4


# Spectrophotometric Method for the Determination of Escitalopram Drug by Ion Pair Complex with Wool Fast Blue Dye in Bulk Dosage and Pharmaceutical Formulation

IJPPR
INTERNATIONAL JOURNAL OF PHARMACY & PHARMACEUTICAL RESEARCH
An official Publication of Human Journals
ISSN 2349-7203
HUMAN

**D Venakateshwarlu[*1], I.E.Chakravarthy[2], Nuzhath Fathima[3], P.Ravindra Reddy[4] and G.Abdul Huq[5]**

*[1,2,5] - Department of Chemistry, Rayalaseema University, Kurnool.*

*[3]- Department of Humanities and Sciences, Al-Habeeb College of Engineering and Technology, Hyderabad.*

*[4] – Department of Chemistry, Sri Krishnadevaraya University, Ananthapur , India.*

**Submission:** 26 October 2016
**Accepted:** 1 November 2016
**Published:** 25 November 2016

**Keywords:** Spectrophotometry, Escitalopram, Wool Fast Blue, Ion-pair Complex, Pharmaceutical formulations

**ABSTRACT**

A simple, sensitive, rapid and selective spectrophotometric method for the micro determination of the anti-depressant drug Escitalopram using Wool Fast Blue (WFB) dye which forms an ion-pair complex in the presence of the buffer solution of pH 1.5. The complex is extracted into chloroform and the absorbances of the extracts are recorded at a wavelength 595 nm. A linear curve is obtained when a graph is drawn between the amount of Escitalopram and the absorbance of the solutions in the range 5 µg/ml to 40 µg/ml. This confirms the obedience of Beer Lambert's law and indicates the suitability of the method for the successful determination of the drug in this range of 5 µg/ml to 40 µg/ml. The molar absorptivity and Sandell sensitivity of the method are $1.0640 \times 10^4$ $lit.mol^{-1}.cm^{-1}$ and 0.03049 $\mu g.ml^{-1}.cm^2$ respectively. The proposed method is successfully applied to evaluate the assay of Escitalopram drug in the bulk dosage form and the pharmaceutical formulations and the results obtained are presented in this paper.

HUMAN JOURNALS

## INTRODUCTION

A survey of chemical and biochemical literature has revealed that several methods such as RP-HPLC [1], Fluorimetric [2] and spectrophotometric [3] have been developed for the determination of Escitalopram drug in pharmaceutical formulations. It is well known beyond apprehension of any kind that drugs and pharmaceuticals play a very significant role in the prevention, control and curing of variety of human diseases. It is very much painful to know about the entry of various spurious and substandard drugs into the market which has a definite adverse effect on the human health. It is with this challenge in mind, the authors have taken up a thorough study to investigate the purity of the various drugs that are released into the market such as Escitalopram. With a view to ascertaining the assay and purity of the drugs, the authors have taken up the simple, sensitive, selective, rapid and versatile instrumental technique of Spectrophotometry. The results obtained in respect of Escitalopram are presented in this communication. Though there are various expensive instrumental techniques such as HPLC, GC, Fluorimetry, NMR, IR etc. available for the assay of drugs. But the authors preferred to employ the technique of Spectrophotometry which is simple, highly versatile and precise.

Escitalopram is chemically (1S)-1-(3-dimethylaminopropyl)-1-(4-fluorophenyl)-3H-2-benzofuran-5-carbonitrile with molecular formula $C_{20}H_{21}FN_2O$. It is an anti-depressant drug and is used in the treatment of body dysmorphic disorder and anxiety. The anti-depressant, anti-obsessive – compulsive and antibulimic actions of Escitalopram are assumed to be linked to its inhibition of CNS neuronal uptake of serotonin. The chemical structure of Escitalopram is as shown below in Fig-1.

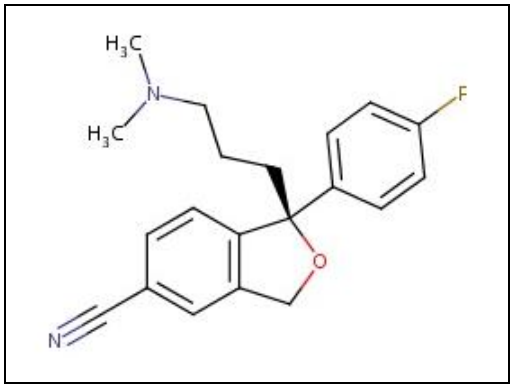



**Fig-1: Structure of Escitalopram**

## MATERIALS AND METHODS

### A. Apparatus: Brief description of instruments employed

**(i) Spectrophotometer**: For spectrophotometric studies Spectronic 1001 plus Spectrophotometer model no. 335002 with battery backup test memory is used. The instrument provides a unique monochromatic design and a variety of microprocess controlled features to get fast and accurate spectrophotometric measurements. It also contains a superior optical system, high-intensity deuterium and tungsten halogen lamps, silica coated steroidal mirrors and Milton Roy own blazed holographic grating which produces exceptional energy and spectral purity. Thus, the instrument has a number of special programming options, functions, test nodes and parameters setups.

**(ii) pH meter**: pH measurements are made using an Elico LI-10 digital pH meter.

### B. Preparation of Reagents and Solutions:

**(i)** Escitalopram solution: 50 mg of pure Escitalopram is dissolved in methanol and the volume of the solution is adjusted to the mark in a 50 ml standard flask with methanol. The stock solution is further diluted to get working concentration of 100 µg/ml.

**(ii)** Buffer solution (pH 1.5): Buffer solution of pH 1.5 is prepared by mixing 289 ml of glycine solution (37.52 gms of glycine and 29.24 gms of NaCl dissolved in 500 ml of distilled water) with 711 ml of 0.1 M HCl.

**(iii)** Wool Fast Blue (WFB) Solution (0.2% w/v): WFB solution is prepared by dissolving 200 mg of the dye in 100 ml of distilled water.

All other chemicals of reagents and solutions used in the present investigation are of AR Grade only.

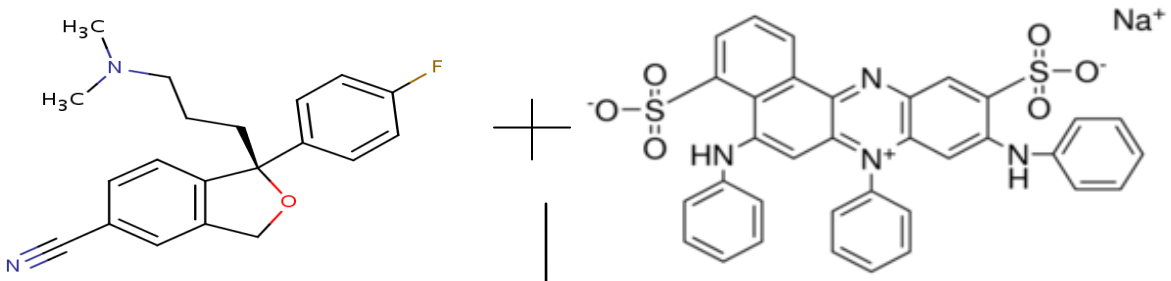
## RESULTS AND DISCUSSION

In the proposed method, the drug Escitalopram is dissolved in water and is treated with WFB at pH 1.5. In the resultant solution, the ion-pair complex is formed which is extracted into chloroform and is suitably employed for the spectrophotometric determination of Escitalopram

with WFB. The probable ion-pair complex formation reaction may be represented as shown below in Fig-2.

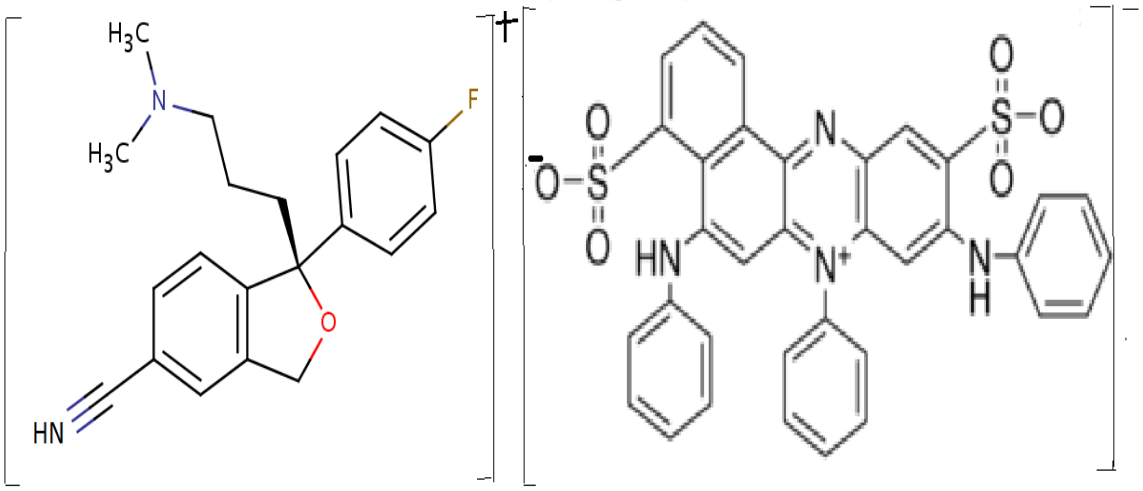
Escitalopram + Wool Fast Blue $\xrightarrow{\text{pH 1.5, } H^+}$ $(ETP)^+ (WFB)^-$

(ETP) (WFB) Extracted into Chloroform Ion Pair Complex



Escitalopram Wool Fast Blue

pH 1.5 , $H^+$ Extracted into Chloroform



$ETP^+$- $WFB^-$ ion-pair complex

**Fig-2: Formation of Ion-pair Complex**

A survey of Chemical Literature has shown that no method is reported for the estimation of Escitalopram by using WFB spectrophotometrically. The various parameters of the present investigation are established as follows:

**Absorption Spectrum of Escitalopram-WFB Complex:** With a view to establish the wavelength of maximum absorbance ($\lambda_{max}$), the absorption spectrum of Escitalopram-WFB Complex is drawn by adopting the following procedure.

2 ml of Escitalopram solution (100 µg/ml), 2 ml of WFB reagent and 3 ml of buffer solution of pH 1.5 are added and the final volume is adjusted to 10 ml with distilled water in a separating funnel. To this, 10 ml of Chloroform is added and the contents are shaken well gently for 5 minutes and then allowed to stand for another 5 minutes so as to separate the aqueous chloroform layers. The colored ion-pair complex is extracted into the chloroform layer and is separated to record the absorbance values in the wavelength range 500 nm to 650 nm against the reagent blank. The results obtained are drawn in the form of a spectrum as shown in Fig-3.

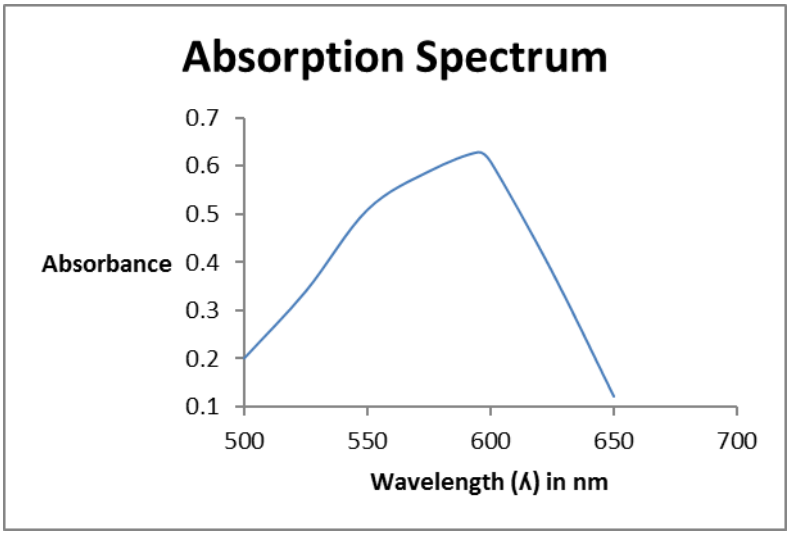



**Fig-3: Absorption Spectrum of Escitalopram–WFB ion-pair complex**

It is seen from the Fig-3 that the absorbance is maximum at 595 nm. Hence the wavelength of 595 nm is fixed for further studies.

**Effect of WFB Concentration:** The effect of WFB on the absorbance of the ion-pair complex is studied by taking varying volumes (x ml) of WFB in a series of separating funnels, keeping fixed volume of Escitalopram (2 ml), buffer solution of pH 1.5 (3 ml), (5-x) ml distilled water to keep the total volume of aqueous layer at 10 ml in each separating funnel. To each of these, 10 ml of chloroform is added, shaken well gently and the complex is extracted into the chloroform layer,

separated and the absorbance values are measured at 595 nm. The data obtained is presented in Table-1.

**Table 1: Effect of WFB Concentration**

Each separating funnel in the series:

2 ml Escitalopram (100 µg/ml) + x ml WFB (0.2% w/v) + 3 ml of buffer pH 1.5 + (5-x) ml distilled water. Total volume of aqueous layer = 10 ml + to this added 10 ml of chloroform, shaken well, complex extracted into chloroform layer, separated and absorbances measured.

$\lambda_{max}$ = 595 nm

| Sr. No. | Volume in ml of WFB Solution | Absorbance |
|---|---|---|
| 1 | 0.5 | 0.545 |
| 2 | 1.0 | 0.559 |
| 3 | 1.5 | 0.586 |
| 4 | 2.0 | 0.621 |
| 5 | 2.5 | 0.601 |
| 6 | 3.0 | 0.587 |
| 7 | 3.5 | 0.564 |
| 8 | 4.0 | 0.549 |

The data in Table-1 shows maximum absorbance at 2 ml of WFB concentration. Hence for all further studies, a volume of 2 ml of WFB is fixed.

**Effect of Volume of Buffer Solution of pH 1.5:** In a series of separating funnels containing 2 ml of Escitalopram (100 µg/ml), 2 ml of WFB (0.2% w/v), x ml of buffer solution of pH 1.5, (6-x) ml distilled water and 10 ml chloroform are added. The contents are thoroughly shaken and the complex is extracted to the chloroform layer which is separated and the absorbances are measured at 595 nm. The results obtained are shown in Table-2.

**Table 2: Effect of volume of buffer solution pH 1.5**

Series of separating funnels: In each funnel

2 ml Escitalopram (100 µg/ml) + 2 ml WFB (0.2% w/v) + x ml of buffer pH 1.5 + (6-x) ml distilled water + 10 ml of Chloroform, shaken well, complex extracted into Chloroform layer, separated and absorbances recorded.

$\lambda_{max}$ = 595 nm

| Sr. No. | Volume in ml of Buffer Solution | Absorbance |
|---|---|---|
| 1 | 0.5 | 0.478 |
| 2 | 1.0 | 0.548 |
| 3 | 1.5 | 0.560 |
| 4 | 2.0 | 0.572 |
| 5 | 2.5 | 0.604 |
| 6 | 3.0 | 0.631 |
| 7 | 3.5 | 0.596 |
| 8 | 4.0 | 0.589 |

It is clear from the data in Tabe-2 that 3 ml of buffer solution of pH 1.5 is necessary to achieve maximum absorbance. Hence for all further experimental studies, a volume of 3 ml of buffer solution of pH 1.5 is maintained.

**Recommended procedure for the determination of the drug Escitalopram: Calibration curve.**

This study actually involves the effect of drug concentration on the absorbance of the ion-pair complex under the established optimal experimental conditions. The recommended procedure is as follows.

Various aliquots (x ml i.e. 0.5 ml to 4.0ml) Escitalopram solution( 100 µg/ml) are transferred into a series of separating funnels followed by the addition of 2 ml of WFB (0.2% w/v), 3 ml of buffer solution of pH 1.5, (5-x) ml of distilled water and 10 ml of Chloroform. The reaction mixture in each funnel is shaken gently well for 5 minutes and allowed to stand for another 5 minutes so as to separate the aqueous and the chloroform layers. The chloroform layer is separated out into which the colored ion-pair complex gets extracted. The absorbance of each

solution is measured at 595 nm against the reagent blank which is prepared in the similar manner omitting the drug solution. The absorbance values obtained are mentioned in Table-3 and Fig-4.

**Table-3: Calibration curve: Estimation of Escitalopram**

Series of separating funnels: In each funnel

x ml Escitalopram (0.5 ml to 4.0 ml Escitalopram Solution 100 µg/ml) + 2 ml WFB (0.2% w/v) + 3 ml of buffer pH 1.5 + (5-x) ml distilled water + 10 ml of Chloroform, shaken well, complex extracted into Chloroform layer, separated and absorbances recorded against reagent blank.

$\lambda_{max}$ = 595 nm

| Sr. No. | Volume of Escitalopram (100µg/ml) in ml X | Amount of Escitalopram in µg/ml | Absorbance |
|---|---|---|---|
| 1 | 0.5 | 5 | 0.164 |
| 2 | 1.0 | 10 | 0.329 |
| 3 | 1.5 | 15 | 0.492 |
| 4 | 2.0 | 20 | 0.631 |
| 5 | 2.5 | 25 | 0.798 |
| 6 | 3.0 | 30 | 0.961 |
| 7 | 3.5 | 35 | 1.125 |
| 8 | 4.0 | 40 | 1.205 |

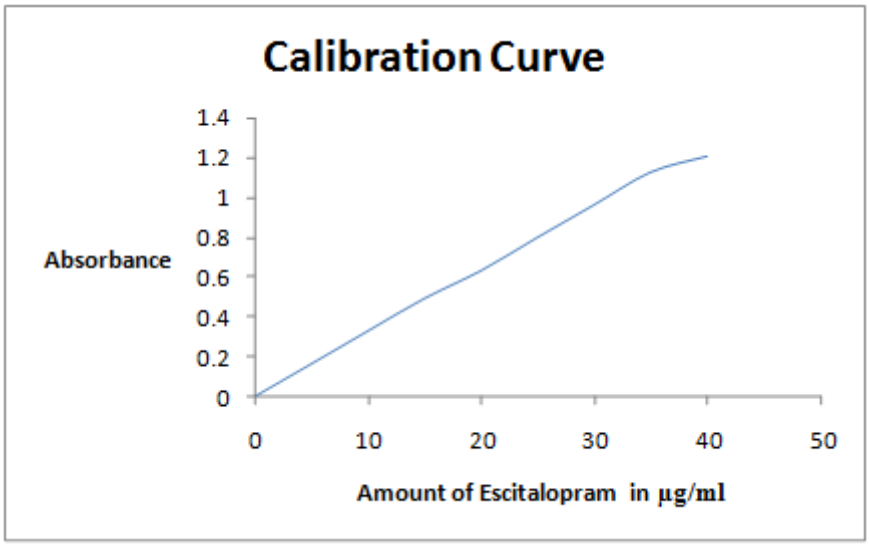



**Fig- 4: Calibration curve– Estimation of Escitalopram**

It is seen from the data in Table-3 and Fig-4 that the absorbance is proportionately linearly increasing with the increase in the amount of Escitalopram indicating the suitability of the method for the quantitative micro determination of Escitalopram up to 40 µg/ml. A linear calibration curve is therefore drawn between the concentration or amount of the drug and the absorbance in the range 5 µg/ml to 40 µg/ml which further confirms the verification of Beer-Lambert's Law. The molar absorptivity and the Sandell sensitivity are calculated as 1.0640 x $10^4$ lit.$mol^{-1}$.$cm^{-1}$ and 0.03049 µg.$ml^{-1}$.$cm^2$.

**Assay of Escitalopram drug in pharmaceutical formulations:**

The proposed recommended procedure for the assay of Escitalopram is applied for its determination in commercial tablets. For analysis of tablet formulations, 20 tablets of Escitalopram are weighed accurately and finely powdered. An accurately weighed portion of the powdered sample equivalent to 50 mg of Escitalopram is taken in a 50 ml volumetric flask containing 25 ml of chloroform, sonicated for 20 minutes. The resultant solution is filtered through Whatman filter paper number 41 into another 50 ml volumetric flask. The filter paper is washed several times with chloroform. The washings are added to the filtrate and the final volume is made up to the mark with chloroform. 5 ml of the filtrate of the sample solution is diluted to 10 ml with chloroform and treated as per the procedure of the calibration curve.

Amount of the drug present in the sample is computed from the respective calibration curves. The results obtained are presented in Table-4.

**Table – 4: Assay of Escitalopram in Tablets**

| Sample | Labelled amount (mg) | Amount found by proposed method ± SD* | Percentage of Label claim | **$t_{cal}$ |
|---|---|---|---|---|
| Tablet I | 20 | 20.02 ± 0.19 | 100.1 | 0.2325 |
| Tablet II | 20 | 19.96 ± 0.40 | 99.8 | 0.2216 |

*Average of 5 determinations based on label claim.

## CONCLUSION

The calibration curve is linear in the range 5 µg/ml to 40 µg/ml of Escitalopram. The standard deviation values are found to be low indicating high accuracy and reproducibility of the method. The calculated 't' values are less than the 't' theoretical values with 4 (n-1 = 5-1) degrees of freedom at 5 % level of significance. This indicates that there is no significant difference between the proposed method and the standard method. Further, there is no effect of additives and excipients such as starch, calcium lactose and glucose in the concentration of those present in general pharmaceutical preparations. Thus, the proposed method can be conveniently adopted for the routine analysis of estimation of Escitalopram in bulk drug samples and pharmaceutical formulations.

## ACKNOWLEDGEMENT

The authors (DV and NF) express their grateful thanks to the authorities of the departments of Chemistry of Rayalaseema University, Kurnool and Sri Krishnadevaraya University, Ananthapur for providing facilities and their constant encouragement throughout the progress of the work.

## REFERENCES

1. Santosh Vilashchand Gandhi, Nilesh Dhyandev Dhavale, Vijay Yeshawantrao Jadhav and Shweta Sadanand Sabnis, Journal of AOAC International, 91(10), 33, (2008).
2. Serebruany, Victor, Malinin, Alex, Dragan, Vadim, Atar, Dan, van Zyl, Louis, Dragan, Anatoly, Clinical Chemistry & Laboratory Medicine, 45(4), 513-520, (2007).
3. Zhangg, Lixia and Xue, Qiang., Zhongguo Yaofang, 18(4), 297-298, (2007).