AMERICAN JOURNAL OF PHARMTECH RESEARCH

**Journal home page:** http://www.ajptr.com/

# UV-Spectrophotometric Determination of Maraviroc in Bulk and Pharmaceutical Dosage Form

**Mastanamma.Shaik[1]*, Haritha Kondayigunta[1], P.Saidulu[1], D.Sindu Priya[1].**

*1. Department of pharmaceutical Analysis, University College of Pharmaceutical Sciences, Acharya Nagarjuna University, Nagarjuna Nagar, GUNTUR, Andhra Pradesh, India-522510.*

## ABSTRACT

The present work describes three simple, precise and economical UVmethods have been developed for the estimation of Maraviroc in bulk and pharmaceutical dosage form. Method A shown the absorbance maxima at 217 nm, method B area under curve method which involves the calculation of integrated value of absorbance with respect to the wavelength between two selected wavelength 212 -222 nm respectively. and in the first order derivative spectra showed sharp peak at 248 nm for method C. The developed methods were founded to be linear in the concentration range of 10-100 µg/ml. The developed methods were validated by following the analytical performance parameters suggested by ICH. All the validation parameters were within the acceptable range. As economical solvent is used, these methods were used for routine analysis of Maraviroc in bulk and pharmaceutical formulation.

**Key words:** Absorption Maxima, Area under curve, Derivative Spectroscopy, Maraviroc, Maraviroc Tablet Dosageform.

*Corresponding Author Email:masthanama.sk@gmail.com

Received 1 February 2014, Accepted12June2014

Please cite this article as: Shaik M *et al.*, UV-Spectrophotometric Determination of Maraviroc in Bulk and Pharmaceutical Dosage Form. American Journal of PharmTech Research 2014.

## INTRODUCTION

Maraviroc is the first CCR5 antagonist and only oral entry inhibitor approved for the treatment of HIV-1infection. These acts as a human immunodeficiency virus type 1 (HIV-1) co receptor. Binding of Maraviroc to this receptor prevents the interaction of HIV-1 gp 120 with CCR5-tropic HIV-1 and there by inhibits the virus from entering the cell [1,2] Maraviroc is chemically 4,4 difluoro N{(1S)3 [exo 3(3 isopropyl 5 methyl 4H 1,2,4 triazol 4 yl)8 azabicyclo[3.2.1] oct 8yl]1 phenyl propoyl} cyclohexane carboxamid . Maraviroc is a white to yellowish or brownish powder with a molecular weight of 514. The molecular formula of Maraviroc C29H41F2N5O and chemical structure is given below (figure.1). Maraviroc is practically insoluble in water slightly soluble in ethanol soluble in methanol, Dimethyl sulfoxide and PEG 400.

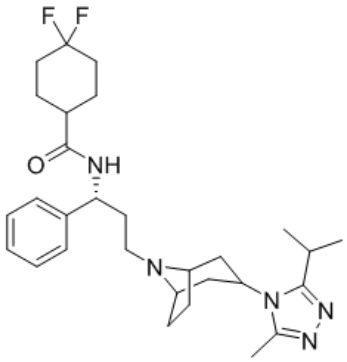


**Figure 1 chemical structure of Maraviroc**

Available literature states few HPLC methods for the estimation of Maraviroc at 210 nm. Though HPLC method is highly sensitive and accurate but cost of analysis is too high. But there is no work in the literature reported about the Derivativemethod and Area under curve method by using UVspectroscopy for the analysis of Maraviroc in pharmaceutical formulation. Hence there is need to develop a simple rapid and economical method for routine analysis of maraviroc. The objective of present study was to develop and validate simple, sensitive, accurate, precise,rapid and economical method for estimation of Maraviroc in bulk and in pharmaceutical formulations as per ICH guidelines

## MATERIALS AND METHODS

### Chemicals and Reagents:

Working standard of Maraviroc (99.85%) was procured as a gift sample from Hetero Labs., Hyderabad, India. Maraviroc tablet dosage form of CELSENTRY 150 mg purchased from local pharmacy. Methanol (AR Grade, Merk, India) and Sodium hydroxide (A.R Grade, Merk, India) distilled water were used for the study.

**Instrumentation:**

UV Visible spectrophotometer Shimadzu model 1800 was employed with spectral band width of 1 nm attach with computer loaded shimadzu UV Pc software (UV probe) version 2.31 and using a pair of 10mm matched quartz cells. Shimadzu Analytical balance was used for the weighing of samples.

**Preparation of standard stock solution and calibration curve**

Working standard Maraviroc 10 mg was weighed accurately and transfer to 10 ml volumetric flask and dissolved in 5 ml of methanol. The flask was shaken for few seconds and volume was made up to the mark with methanol to get the concentration of 1000 mcg/ml. from this solution series of aliquots with concentration range of 10- 100 µg/ml were prepared by using 0.1 N NaoH as a solvent to construct the calibration curve. Linearity for detector response was observed in the concentration range of 10-100 µg/ml for method A, method B and method C.

**Method A: Absorption Maxima Method**

By appropriate dilution of stock solution and scanned in spectrum mode from 200 -400nm, the maximum wavelength 217 nm was selected for the analysis. The calibration curve for Maravoroc was plotted in the concentration v/s absorbance as shown in Figure 2 and. From this regression equation was calculated. This equation was used to estimate the Maravorocin tablet dosage forms

**Method B: Area under Curve Method**

Area under curve (AUC) method involves the calculation of integrated value of absorbance with respect to the wavelength between two selected wavelength 212 -222 nm. Area calculation processing item calculates the area bound by the curve and the horizontal axis. The horizontal axis is selected by entering the wavelength range over which the area has to be calculated. The wavelength range is selected on the basis of repeated observations so as to get the linearity between the area under curve and concentration as shown in Figure 3. From this regression equation was calculated. This equation was used to estimate the Maravoroc in tablet dosage forms

**Method C: First order derivative method**

It involves the conversation of a normal spectrum to its zero, first, second or higher derivative spectrum. In derivative spectrophotometry, spectra are obtained by plotting the first or a higher order derivative of absorbance with respect to wavelength as a function of wavelength. Often, these plots reveal spectral details that is lost in an ordinary spectrum. In addition, concentration measurements of an analytein the presence of interference or of two or more analytes in a mixture can sometimes be made more easily or accurately using derivative methods. In this method, 20µg/ml solution of Maraviroc was prepared by appropriate dilution of standard stock solution and

scanned from 200-400 nm. The absorption spectra thus obtained were derivatized from zero to second order. First order derivative spectra of the drug showed a sharp peak at 248 nm, which was selected for its quantitation. The concentration ofMaraviroc present in the test solution was determined against the calibration curve as shown in Fig no 3 in quantitation mode.

**Analysis of marketed formulations**

For estimation of Maraviroc in tablets formulations by these methods 20 tablets of marketed brand of Celsentry were weighed and triturate to fine powder. Amount of powder equivalent to 50 mg drug was taken and dissolved in 10 ml of methanol and made up to the mark with methanol in 50 ml volumetric flask (1000 µg/ml). It was filtered with whatmann filter paper no. 41 and from that stock solution further dilution were made with 0.1N NaoH to get required concentration. In method A, the concentration of Maraviroc was determined by absorbance of sample solution at 217 nm. For method B, the concentration of Maraviroc was determined by measuring the absorbance of sample solution in the wavelength range of 212-222 nm. In method C, the concentration of Maraviroc was determined by measuring the amplitude difference at 248 nm. Results of tablet analysis were shown in Table 1. The assay procedure was repeated six times

**VALIDATION**

The methods were validated according to ICH guidelinesQ2(R1) to study linearity, accuracy and precision by using above three methods.

**Linearity**

A linear relationship was found for the three methods between the absorbance and the concentration of maraviroc in the range of 10-100 µg/ml. The correlation coefficient for these methods A, B, C were 0.996, 0.998, 0.999 indicating linearity ($r^2$> 0.999) (Table 2)

**Precision**

The precision of the method was expressed in terms of % relative standard deviation (% RSD). The % RSD values for three methods A, B, C found to be less than 2 for intraday and inter day precision, the precision results showed good reproducibility. The results are expressed in Table 3.

**Accuracy**

Accuracy for the methods A, B, C were established at 80, 100, 120% levels by the addition of standard drug of Maraviroc to pre-analyzed samples. The results were given in Table no.4.

**Limit of Detection and Limit of Quantitation:**

LOD and LOQ values were calculated from the data obtained from the linearity studies. The slope of the linearity plot was determined. For each of the six replicate determination, Y intercept and its standard deviation was computed. From these values, the parameters Limit of Detection (LOD)

and Limit of Quantitation (LOQ) were determined on the basis of response and slope of the regression equation. The results were given in Table .2

## RESULTS AND DISCUSSION

The methods discussed in the present work provide a convenient and accurate way for the analysis of Maraviroc in bulk and in pharmaceutical dosage form. The absorbance Maxima of Maraviroc was found at 217 nm (Method A) and the wavelength range for area under curve (Method B) was 212-222 nm. The first order derivative spectroscopy method sharp peak at 248 nm (Method C) were selected for the analysis. linearity for detector response was observed in the concentration range of 10-100 µg/ml for Method A, Method B, Method C. standard deviation and coefficient of variance for six determinations of tablet sample using all the methods were to be less than ± 2.0. The validation of proposed methods were further confirmed by recovery studies, the %recovery values vary from 98- 102%. Based on results obtained it was found that the proposed methods were accurate, precise, reproducible and can be employed for routine quality control of Maraviroc tablet dosage form.

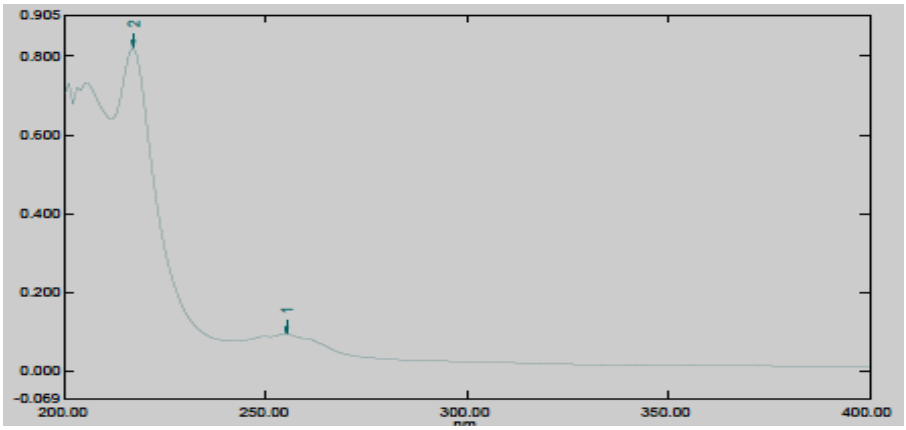


**Figure 2: Absorption Maxima Spectrum of Maraviroc**

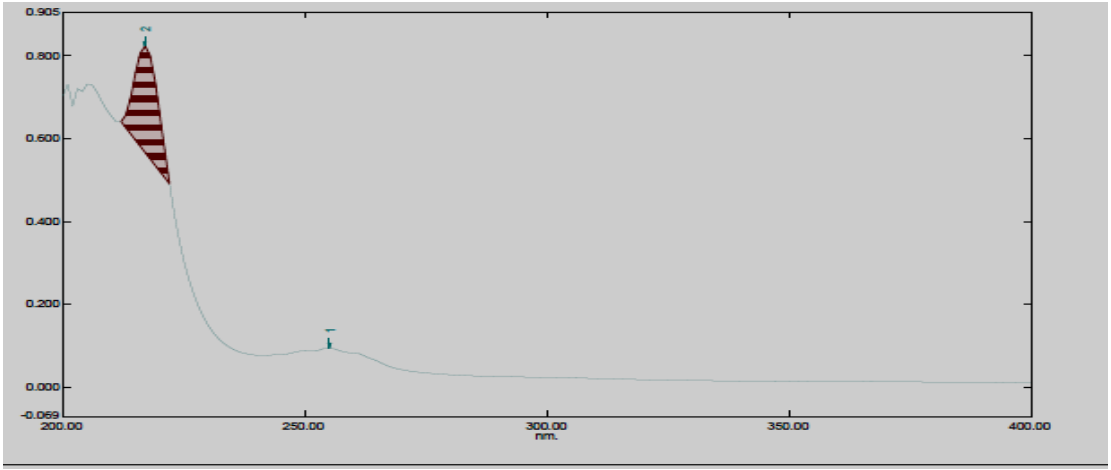


**Figure 3: UV-Spectrum of Maraviroc indicating AUC**

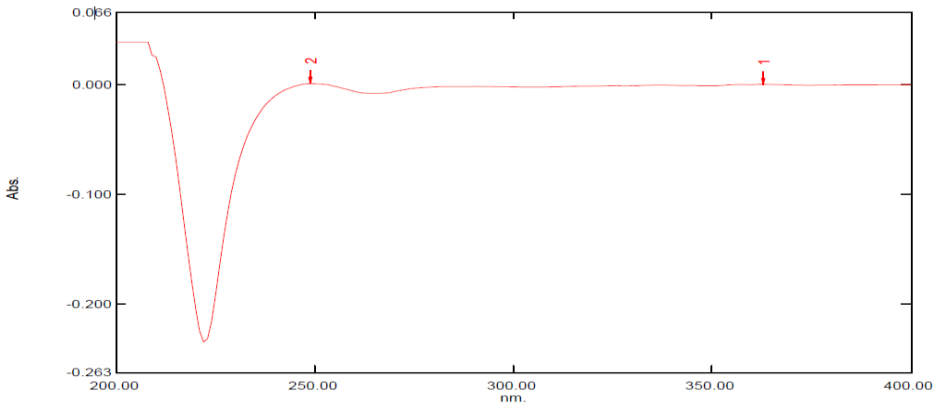


**Figure 4: First order derivative spectrum of Maraviroc**

**Table.1: Result of Marketed Formulation Analysis**

| Proposed methods | Label claim (mg) | Mean | Std.deviation | %RSD |
|---|---|---|---|---|
| A | 150 | 98.07 | 0.25 | 0.06 |
| B | 150 | 99.28 | 0.03 | 0.03 |
| C | 150 | 99.71 | 0.01 | 0.01 |

**Table 2: Optical characteristics of Maraviroc**

| S.No | Parameter | Method A | Method B | Method C |
|---|---|---|---|---|
| 1 | Linearity (µg/ml) | 10-100 | 10-100 | 10-100 |
| 2 | Linearity Equation | Y= 0.0104x + 0.002 | Y= 0.0532x + 0.05 | Y= 0.0003x -0.0002 |
| 3 | Slope | 0.0104 | 0.0532 | 0.0003 |
| 4 | Intercept | 0.002 | 0.05 | 0.0002 |
| 5 | Correlation coefficient ($r^2$) | 0.996 | 0.998 | 0.999 |
| 6 | LOD (µg/ml) | 0.092 | 0.047 | 0.122 |
| 7 | LOQ (µg/ml) | 0.275 | 1.441 | 0.400 |

**Table 3: Intra day and Inter day data of Precision of Maraviroc**

| Method | Label Claim (mg) | Intraday precision % RSD (n=3) | Interday precision %RSD (n=3) | |
|---|---|---|---|---|
| | | | Day I | Day II |
| A | concentration taken | 0.55 | 0.49 | 0.52 |
| B | 20(µg/ml) | 0.62 | 0.52 | 0.60 |
| C | | 0.83 | 0.78 | 0.80 |

**Table 4 : Recovery data of Maraviroc**

| Method | Test Concentration(µg/ml) | Level of % recovery | Amount added (µg/ml) | Amount found (µg/ml) (n=6) | % Analytical Recovery (n=6) |
|---|---|---|---|---|---|
| Method | 40 | 80 | 32 | 70.96 | 99.6 |
| A | 40 | 100 | 40 | 78.65 | 98.56 |
| | 40 | 120 | 48 | 87.30 | 100.12 |
| Method | 40 | 80 | 32 | 70.85 | 98.91 |
| B | 40 | 100 | 40 | 79.23 | 101.01 |
| | 40 | 120 | 48 | 87.93 | 100.96 |
| Method | 40 | 80 | 32 | 70.96 | 99.49 |
| C | 40 | 100 | 40 | 77.95 | 96.23 |

| | | | | |
|---|---|---|---|---|
| 40 | 120 | 48 | 86.95 | 99.89 |

## CONCLUSION

The proposed UV spectrophotometric methods were found to be simple, sensitive, accurate, precise and economical and can be used in the determination of Maraviroc in bulk and pharmaceutical dosage forms in a routine manner.

## ACKNOWLEDGEMENT

The authors express their gratitude to Hetero Pharmaceuticals, Hyderabad for providing gift sample of working standard. Special thanks to Dr. A.Pramela Rani Principal, ANU college of pharmaceutical sciences for providing excellent laboratory facility to carry out this research work.

## REFERENCES

1. www.drugbank.ca (maraviroc)
2. www.rxlist.com (maraviroc)
3. L. Satyanarayana, S.V. Naidu, M. NarasimhaRao, C. Ayyanna, and Alok Kumar; Research Journal of Pharmaceutical Dosage Forms and Technology, 2011; 3(5):230.
4. United States Pharmacopeia USP 34-NF 29, (2011)
5. KalyanChakravarthy. V and GowrisankarD.Development and validation of RP-HPLC methods for Maraviroc in bulk and its pharmaceutical formulation. Int. Pharma. Res. Development 2011; 3(10): 72-79