

# Derivative Spectrophotometric Estimation of Minocycline Hydrochloride in Bulk and Pharmaceutical Dosage Forms

Popat B. Mohite<sup>\*1</sup>, Ramdas B. Pandhare<sup>1</sup>, Shantaram G. Khanage,  
<sup>1</sup>Vaidhun H. Bhaskar<sup>2</sup>

<sup>1</sup>MES College of Pharmacy, Sonai, Ahmednagar, Maharashtra, India

<sup>2</sup>MP Patel College of Pharmacy, Kapadwanj, Gujrat, India.

*\*Corres.author: mohitepb@rediffmail.com*

*Mob no.-09890520854*

*Fax No.02427 230948*

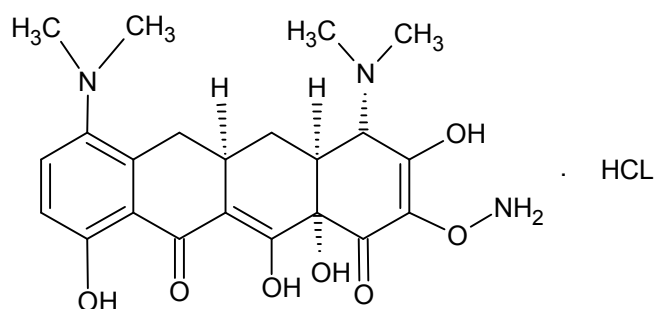
**Abstract:** A simple, precise and economical spectrophotometric method have been developed for the estimation of Minocycline Hydrochloride in bulk and pharmaceutical formulations. Minocycline Hydrochloride shows a sharp peak at 283.0 nm in first order derivative spectrum with  $n = 1$ . The drugs follows Beer-Lambert's law in the concentration range of  $1-10 \mu\text{g mL}^{-1}$  in both the methods. Result of the analysis was validated statistically and found to be satisfactory.

**Key Words:-** Minocycline Hydrochloride, Derivative spectroscopy.

## Introduction

Minocycline Hydrochloride is a Chemically (4S, 4aS, 5aR, 12aS)-4,7-bis (dimethylamino)-1,4,4a,5,5a,6,11,12a-octahydro-3,10,12,12a-tetrahydroxy-1,11-dioxonaphthacene-2-carboxamide hydrochloride monohydrate. It is clinically useful as a broad-spectrum antibiotic. Minocycline Hydrochloride is a bacteriostatic and acts interfere with protein biosynthesis by binding to rRNA in the 30S subunit of ribosome. It is official in British pharmacopoeia and United State pharmacopoeia It is listed in The Merck Index and Martindale.<sup>1,2</sup> Literature survey reveals that only few HPLC<sup>3,4,5</sup> and Visible Spectrophotometric<sup>4</sup> methods are reported for the determination of Minocycline Hydrochloride. The objective of the present work is to develop new spectrophotometric methods for estimation of Minocycline Hydrochloride in bulk and formulation with good accuracy, simplicity, precision and economy.<sup>7</sup>

Figure 1-Structure of Minocycline Hydrochloride



## Experimental

### Materials and Methods

Pure sample of Minocycline Hydrochloride was obtained from Alkem Lab. Ltd., Mumbai as a gift sample and capsule of Minocycline Hydrochloride (50 mg) procured from local market. Methanol used as

solvent, JASCO V-630 UV/VIS spectrophotometer was used with 1cm matched quartz cells. Accurately about 100mg of the pure drug was weighed and dissolved in sufficient quantity of methanol and volume made up to 100ml with methanol to give standard stock solution (1 mg/ml). Aliquots of standard stock solution were pipette out and suitably diluted with methanol to get final concentration of 1-10  $\mu\text{g mL}^{-1}$  of standard solution. The solution were scanned in the spectrum mode from 450 nm to 190 nm wavelength range and the first order derivative spectra were obtained at  $n=1$  a sharp peak was obtained at 283.0 nm (Figure-2).

The absorbance difference at  $n=1$  ( $dA/d\lambda$ ) was calculated by the inbuilt software of the instrument which is directly proportional to the concentration of the standard solution. A calibration curve was plotted taking the absorbance difference ( $dA/d\lambda$ ) against the concentration of the standard solutions. The method was applied for the sample solution of known concentration and was found to be satisfactory for analysis of tablet formulation. Optical characteristics of azathioprine indicated in Table 1.

#### Analysis of Pharmaceutical Dosage Forms

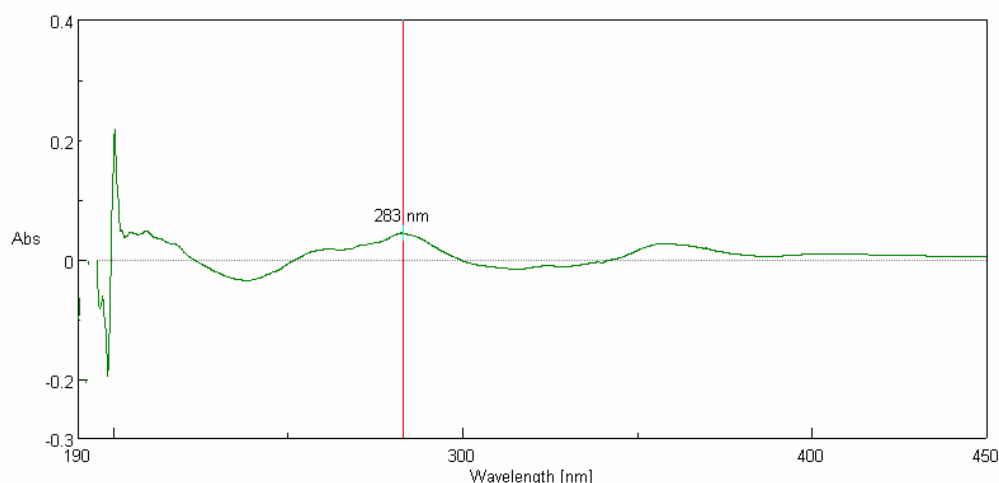
To determine the content of Minocycline Hydrochloride capsules (label claim: 50 mg of

Minocycline Hydrochloride) twenty capsules were weighed, their average weight determined. The weight equivalent to 50 mg of Minocycline Hydrochloride was taken and amount of powder was dissolved in methanol by stirring for 30 min. The excipients were separated by filtration. After filtration, an appropriate amount of internal standard was added and diluted up to mark with methanol. Appropriate aliquots were subjected to above methods and the amount of Minocycline Hydrochloride were determined. The results are reported in Table 2.

#### Recovery studies

To check the accuracy of the developed methods and to study the interference of formulation additives, analytical recovery experiments were carried out by standard addition method, at 80, 100 and 120 % level. From the total amount of drug found, the percentage recovery was calculated. The results are reported in Table 3.

**Figure-2 First Derivative Spectrum of Minocycline Hydrochloride**



**Table 1: Optical Characteristic And Other Parameters**

| Parameters                                 | Derivative spectroscopy |
|--------------------------------------------|-------------------------|
| $\lambda$ max (nm)/wavelength range (nm)   | 283.0                   |
| Beer-Lambert's range $\mu\text{g mL}^{-1}$ | 4-24                    |
| Coefficient of correlation (r)             | 0.9989                  |
| a. Slope (m)                               | 0.0187                  |
| b. Intercept (c)                           | 0.5242                  |

**Table 2: Estimation Of Minocycline Hydrochloride In Formulation.**

| Method                  | Capsule | Label claim(mg) | Amount Found(mg) | %     | S.D*  | S.E    |
|-------------------------|---------|-----------------|------------------|-------|-------|--------|
| Derivative spectroscopy | C1      | 50              | 49.96            | 99.92 | 0.037 | 0.0164 |

\*Each value is average of six estimations

**Table 3: Recovery Studies**

| Derivative spectroscopy method |           |        |
|--------------------------------|-----------|--------|
| Excess drug                    | *Recovery | %RSD   |
| 80                             | 99.83     | 0.2953 |
| 100                            | 99.72     | 0.2026 |
| 120                            | 100.58    | 0.9702 |

\*Recovery is mean of three estimations

## Result And Discussion

The developed method for estimation of Minocycline Hydrochloride in capsule dosage form were found to be simple, accurate and reproducible. Beer-Lambert's law obeyed in the concentration range of 1-10  $\mu\text{g mL}^{-1}$ . The values of standard deviation were satisfactory and recovery studies were close to 100 %. It also eliminates the interference caused by the excipients and the degradation product present, if any, in the formulation. Hence these methods can be useful in the routine analysis of Minocycline Hydrochloride in bulk drugs and formulations.

## Acknowledgement

The authors are thankful to Alkem Lab. Ltd., Mumbai, for providing drug samples and Principal MES College of Pharmacy, Sonai for providing facilities to carry out this work.

## References

- [1] United State Pharmacopoeia NF ,The official compendia of standards,Asiain Edn, 1238-1240,2003.
- [2] Sean,C.,Sweetman,Eds:In.,Martindale,The Complete Drug Reference London, Edn.33,224,2002.
- [3] T.L.Lemke, D.A.Williams, V.F.Roche, S.W.Zito, Foyes Principle of Medicinal Chemistry, Edn 6,1073-76,2002.
- [4] Hoshino Mitsuru, Mori Itsuo, Motomatsu Miho, Nakatani Kenzo, Yamaguchi Takako, Fujita Yoshikazu . Spectrophotometric determination of minocycline hydrochloride with p-carboxyphenylfluorone and titanium (IV). Journal of Bunseki Kagaku, 50,547-551,2002.
- [5] Marcus V. F. Araujo, Demian R. Ifa, Wellington Ribeiro, Maria E. Moraes, Manoel O.

Moraes and Gilberto de Nucci. Determination of minocycline in human plasma by high-performance liquid chromatography coupled to tandem mass spectrometry: application to bioequivalence study. Journal of Chromatography B: Biomedical Sciences and Applications 75, Issue 1-2, 1-7, 2001

[6] Milena Colovic and Silvio Caccia, Liquid chromatographic determination of minocycline

in brain-to-plasma distribution studies in the rat, Journal of Chromatography B

791; Issue 1- 2, 337-343, 2003..

[7] Vieweg Verlag,. Simple Liquid Chromatographic Determination of Minocycline in Brain and Plasma. Journal of Chromatographia 65, 277-281, 2007.

[8] Stenlake J B, Backett A H , Practical pharmaceutical Chemistry, Edn 4, Part 2, 81, 1997.