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## Research Article

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### Analytical Method Development and Validation of Naratriptan in Bulk and Tablet Dosage form using UV Spectrophotometer

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#### ABSTRACT

**Objective:** To develop and validate an analytical method for quantitative determination of Naratriptan in bulk and tablet dosage form. **Results:** The Naratriptan shows absorption maxima at 224.20nm and obeyed Beer's law in the range of 2-10µg/ml. The limit of detection and limit of quantitation were 9.75 and 29.55µg/ml respectively. Percentage recovery of Naratriptan for the proposed method ranged from 98.1% to 102.2%. **Conclusion:** It was concluded that the proposed method is simple, easy to apply, economical and used as an alternative to the existing Spectrophotometric method for the routine analysis of Naratriptan in pharmaceutical formulations.

**Keywords:** Naratriptan, UV Spectrophotometer.

#### ARTICLE INFO

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#### 1. Introduction

**Naratriptan** N-methyl-2-[3-(1-methylpiperidin-4-yl)-1H-indol-5-yl] ethane sulfonamide It is a selective agonist of serotonin (5-hydroxytryptamine; 5-HT) type 1B and 1D receptors. It is structurally and pharmacologically related to

other selective 5-HT<sub>1B/1D</sub> receptor agonist. Naratriptan has only a weak affinity for 5-HT<sub>1A</sub>, 5-HT<sub>5A</sub>, and 5-HT<sub>7</sub> receptors and no significant affinity or pharmacological activity at 5-HT<sub>2</sub>, 5-HT<sub>3</sub> or 5-HT<sub>4</sub> receptor subtypes or at

alpha1-, alpha2-, or beta-adrenergic, dopamine1;, dopamine2; muscarinic, or benzodiazepine receptors. This action in humans correlates with the relief of migraine headache. In addition to causing vasoconstriction, experimental data from animal studies show that Naratriptan also activates 5-HT<sub>1</sub> receptors on peripheral terminals of the trigeminal nerve innervating cranial blood vessels, which may also contribute to the antimigrainous effect of Naratriptan in humans. Three distinct pharmacological actions have been implicated in the antimigraine effect of the triptans: (1) stimulation of presynaptic 5-HT<sub>1D</sub> receptors, which serves to inhibit both dural vasodilation and inflammation; (2) direct inhibition of trigeminal nuclei cell excitability via 5-HT<sub>1B/1D</sub> receptor agonism in the brainstem and (3) vasoconstriction of meningeal, dural, cerebral or pial vessels as a result of vascular 5-HT<sub>1B</sub> receptor agonism.

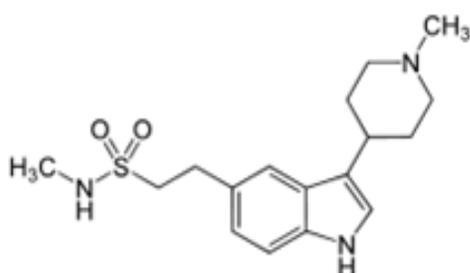


Figure 1: Naratriptan

## 2. Materials and Methods

### Apparatus

#### Spectrophotometric conditions

Spectral and absorbance measurements were carried out by using UV- Visible spectrophotometer model T60, with spectral bandwidth of 2.0 nm and wavelength accuracy of  $\pm 0.5$  nm. Pair of 10 mm quartz cells were used for the absorbance measurements connected with UVWIN version 5.2.0 software.

#### Preparation of standard stock solutions of Naratriptan:

Standard Naratriptan 10mg was weighed and transferred to a 100ml volumetric flask and dissolved in water. The flask was shaken well, from this take 1ml into a 10ml volumetric flask and make up with water up to mark to obtain 10 $\mu$ g/ml solution.

#### Preparation of test Solution (Analysis in tablets):

Ten tablets of formulation, (Naratrex), Sun Pharma, India, were weighed and finely powdered. The powder equivalent to 10mg of Naratriptan was accurately weighed and transferred to volumetric of 100ml capacity, make up volume with water and sonicated for 10mins. From the above solution pipette out 1ml and diluted to 10ml with water to give a solution of 10 $\mu$ g/ml and used for estimation of Naratriptan.

## 3. Results and Discussion

Naratriptan was analyzed by developed by UV Spectrophotometric method in tablets. The UV spectrum shows absorption maxima at 224.20nm. The calibration curve showed linearity over a concentration range from 2-10.0 $\mu$ g/mL, which follows the Beer and Lambert's law. The Journal of Pharmaceutical and Biomedical Analysis Letters

correlation coefficient of the curve obtained with linear regression method was 0.998. The linear regression data for the calibration plot is indicating of a good linear relationship between absorbance and concentration over a wide range. The correlation coefficient was indicative of high significance. The low value of intercept of the ordinate showed the calibration plot did not deviate from linearity. The LOD and LOQ values were found to be 9.75 $\mu$ g/mL and 29.55 $\mu$ g/mL, respectively. LOD and LOQ were found to be in microgram level indicating the sensitivity of the method. The method was also found to be robust and rugged as indicated by the %RSD values which are less than 2%. The recovery was assessed from three replicate determinations of three different solutions containing 8.0, 10.0, 12.0 $\mu$ g/mL. The absolute means obtained were 98.1, 99.7, and 102.2% respectively. It is evident that the method is accurate within the desired range. The precision of the method was checked by carrying out six independent assays of Naratriptan test samples against a working standard. Intermediate precision was checked by analyzing the samples by two different analysts using same instrument. The lower percentage RSD (<2.0%) values shows the method is more precise. The developed method was applied to the quantification of Naratriptan in tablets available in local market. It can be seen that, the results obtained by proposed method was very much similar to that of established methods.

### Method development

Various solvents were selected for the solubility studies and found that Naratriptan was soluble in the following solvents: water, acetonitrile, and methanol. In the present investigation Water was selected as solvent. Appropriate dilutions were prepared using standard stock solution and the solution was scanned in the wavelength range of 200-400nm. The absorption maximum was found at 224.20nm. Appropriate volumes of aliquots from standard solution were transferred to different volumetric flasks of 10ml capacity. The volume was adjusted to mark with water to obtain concentrations of 2, 4, 6, 8 and 10 $\mu$ g/ml. Absorbance value of each solution against water as a blank was measured at 224.20nm.

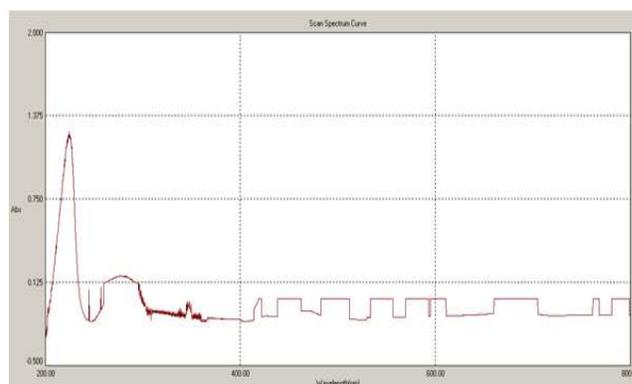


Figure 2: UV spectrum of Naratriptan standard

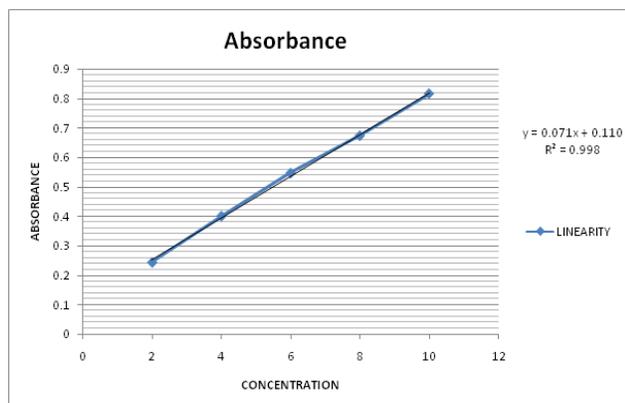
### Method validation

After development of method, validation of the method for simultaneous estimation of Naratriptan was performed in accordance with ICH guidelines (International Conference on Harmonization (ICH) 2000) which include Linearity,

Accuracy, Precision, LOD and LOQ, Ruggedness and Robustness.

### Linearity

The spectrophotometry method showed good linearity for Naratriptan in the range of 2-10 $\mu$ g/ml with regression equation, correlation coefficient and slope are respectively.



**Figure 3:** Calibration curve for Naratriptan at 224.20nm

### Accuracy:

The percentage recovery greater than 98% shows that the method is free from the interference of excipients used in the formulation.

### Precision

The method was found precise on intraday and inter day basis as the average %RSD value for the determination of Naratriptan was as shown in (table: 09, 10, 11, and 12). The %RSD was found to be less than 2, the high precision of method.

### Ruggedness:

Ruggedness is also called by the name intermediate precision. The intermediate precision is the precision obtained by the assay is performed by multiple analysts (analyst-1 and analyst-2) in same laboratory. Using the 6 different concentrations like inter day and intraday precision. Intermediate precision results are used to identify which of the factors contribute significant variability to the final result.

**Limit of detection (LOD) and limit of quantitation (LOQ):** The LOD and LOQ of Naratriptan were determined by calculating the signal-to noise (S/N) ratio of 3:1 and 10:1, respectively according to International Conference on Harmonization guidelines. LOD values for Naratriptan were found to be 9.75 $\mu$ g/ml. LOQ values for Naratriptan was found to be 29.55 $\mu$ g/ml.

**Table15:** Summary of validation parameters

Parameter	Result
Linearity indicated by correlation coefficient	0.998
Precision indicated by % RSD	0.922
Accuracy indicated by % recovery	100%
Limit of detection	9.75 $\mu$ g/ml
Limit of quantification	29.55 $\mu$ g/ml
Range	2-10 $\mu$ g/ml
Linear regression equation	Y=0.071x

Ruggedness indicated by % recovery	98.2%
Assay indicated by % recovery	100.1%

## 4. Conclusion

The present results provide clear evidence that the proposed method can be successfully used for determination of drug content in marketed formulations.

## 5. Acknowledgement

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