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Development, Validation and Stability Study of UV Spectrophotometric Method for Determination of Tenoxicam in Bulk and Pharmaceutical Dosage Forms

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Abstract: ***A simple, specific and economic UV spectrophotometric method has been developed using as a solvent 0.1N NaOH: Methanol (8:2) to determine the Tenoxicam in bulk and pharmaceutical dosage formulations. The quantitative determination of the drug has been carried out at a predetermined $\lambda_{max}$of 370nm, it was proved linearin the range 2-12 µg/mL and exhibited good correlation coefficient (R^2=0.996) and excellent mean recovery (98-100.09%). The method was validated statically and by recovery studies for linearity, precision, repeatability and reproducibility as per ICH guideline. The obtained results proved that the method can be employed for the routine analysis of daclatasvirin bulk as well as in the commercial formulations.***

Keywords: Tenoxicam, UV Spectroscopy, Validation, Stress Studies

1. Introduction

Validation of an analytical method is the process by which it is established, by laboratory studies, that the performance characteristics of the method meet the requirements for the

Intended analytical applications.

Tenoxicam chemically 4-hydroxy-2methyl-n-(pyridinyl-2-yl)-2h-thieno [2, 3-e]-1, 2thiazine-3-carboxamide 1, 1-dioxide, is a Non steroidal anti inflammatory drug [1]. It is used to relieve inflammation, swelling, stiffness, and pain associated with rheumatoid arthritis, osteoarthritis, ankylosing spondylitis .It is official in BP [2]

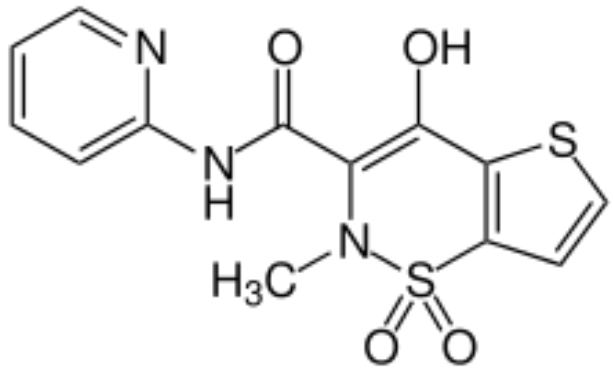

Figure 1: Chemical Structure of Tenoxicam

Literature survey reveals LC-MS [3], HPLC [4-10], spectrophotometric [10-15] methods for the estimation of Tenoxicam from pharmaceutical formulation. A Shimdzu double beam UV/Vis spectrophotometer model 1800 with 1 cm matched quartz cells was used for absorbance measurement.

2. Materials and Methods

Materials and Methods

Instruments
A Shimadzu UV–visible spectrophotometer (UV1800, Shimadzu Corporation, Kyoto, Japan)was used for all absorbance measurements with matched quartz cells.

2.1 Materials

All chemicals and reagents were of analytical grade.Tenoxicam in the form of powder withcertificate of analysis was provided by Ramdev Chemical Pvt Ltd Thane. Pharmaceuticalgrade excipients were obtained from Pharmaceutical Technology Lab. of Maharashtra.

2.2 Determination of wavelength of maximum absorption

A standard stock solution of Tenoxicam (100 µg/mL) was prepared using diluents to further obtain 10 µg/mL. An UV spectroscopic scanning (200-400 nm) was carried out with final diluted solution to determine λ_{max} for the detection of Tenoxicam using diluents as a blank.

2.3 Linearity and Range

For linearity study, six solutions at different concentrations (2, 4, 6, 8, 10 and 12 mg/mL) were prepared using six different aliquots of stock solution, and the obtained data were used for the linearity calibration plot. Limit of detection (LOD) and limit ofquantification (LOQ) for the assay were also calculated

2.4 Intra-day precision (repeatability) and inter-day precision study (intermediate precision)

Tenoxicam tablets were finely powdered and the sample stock solution of 10mg/mL was prepared followingthe same dilution pattern of stock solution. Three different aliquots of stock solution were then diluted to 10 mL to obtain the concentrations of 4, 6 and 8 mg/mL. This procedure was repeated in the following days.

2.5 Stability study

Samples prepared for repeatability study were preserved for 24 h at room temperature and analyzed on the following day to test for short-term stability.

2.6 Accuracy/recovery study

This study was carried out using pre-formulated granules containing pure Tenoxicam and common excipients. Calculation was done from the label claim and the average weight of the final product. Previously used dilution pattern was followed for the granules to obtain three concentrations—80%, 100% and 120% of reference solution.

2.7 Specificity in the presence of excipients

The test for the specificity was carried out using only excipients. Spectra for placebo granules, blank, and sample were compared. Secondly the specificity was determined by subjecting the sample solution to accelerated degradation by heat (60 °C) for 48 h in order to verify that none of the degradation products interfered with the quantification of the drug.

2.8 Assay of content of Tenoxicam in selected marketed brands

Market brands of Tenoxicam tablet from different manufacturers were randomly selected and analyzed using the newly developed and validated method. Sample solutions of each brand (10 mg/mL) were also prepared and assayed for content of Tenoxicam against the standard. The content of Tenoxicam in the marketed brands was determined using standard calculations.

2.9 Stress degradation studies

1) **Photolytic Degradation**
   Specific amount of drug Tenoxicam was weighed accurately & putted into the UVchamber for three days. After three days 10mg drug was weighed and madestock solution (100µg/mL) with diluents. Then an appropriate concentration (10 mg/mL) wasprepared & absorbance was measured in UV spectrophotometer.
2) **Thermal Degradation**
   Drug was taken in a Petri dish which was previously cleaned & dried then was put it into the oven for 48 hrs then it was taken out & weighed 10mg drug was weighed and made stock solution (100µg/mL) with diluents. Then an appropriate concentration (10µg/mL) wasprepared & absorbance was measured in UV spectrophotometer.
3) **Acid Degradation**
   0.01N HCl was taken in a 10 ml volumetric flask then accurately weighed 10mg drug Tenoxicam was dissolved in it. Then the solution was refluxed for 4 hrs then from this solution an appropriateconcentration (10µg/mL) wasprepared using diluents & absorbance was measured in UV spectrophotometer.
4) **Alkali Degradation**
   0.01N NaOH was taken in a 10 ml volumetric flask then accurately weighed 10mg drug Tenoxicam was dissolved in it. Then the solution was refluxed for 4 hrs then from this solution an appropriateconcentration (10µg/mL) wasprepared using diluents & absorbance was measured in UV spectrophotometer.
5) **Oxidation with H_2O_2**
   3% H2O2solutionwas taken in a 10 ml volumetric flask then accurately weighed 10mg drug Tenoxicam was dissolved in it. Then the solution was kept in dark for 4 hrs then from this solution anappropriate concentration (10µg/mL) was prepared using diluents & absorbance was measured in UV spectrophotometer.

3. Results and Discussion

3.1 Method Development and Optimization

Tenoxicam is almost insoluble in aqueous medium and sparingly soluble in organic solvents like methanol. And dissolved in 0.1N NaOH. During the development phase, the use of a few milliliters of methanol with 0.1N NaOH as the diluents resulted in preferable outcome in UV analysis. The solvent composition was optimized to Methanol (2): 0.1N NaOH (8). The pre-determined wavelength of maximum absorption (λ_{max}) was 370 nm. (Fig. 2)

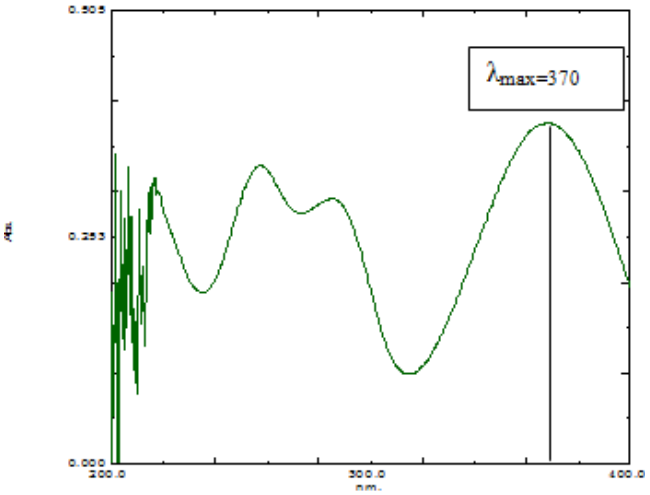

Figure 2: UV Spectrum of Tenoxicam

3.2 Method validation

3.2.1 Linearity and range

The calibration curve obtained was evaluated by its correlation coefficient. The absorbance of the samples in the range of 2.0–12.0 mg/mL was linear with a correlation coefficient (R^2) greater than 0.996 (Table 1). The LOD and

LOQ were calculated as 0.871mg/mL and 2.641L respectively.

Table 1: Linearity data

Concentration µg/ml	Absorbance
2	0.022
4	0.122
6	0.257
8	0.422
10	0.547
12	0.667

3.2.2 Intra-day and inter-day precision

The intra-day and inter-day precision study (Table 2) of the developed method confirmed adequate sample stability and method reliability where all the RSDs were below 2%.

Table 2: Intra-day and inter-day precision determined for three different concentrations of Tenoxicam (n=3).

Concentration µg/mL	Intra-day precision			Inter-day precision		
	Absorbance measured	RSD (%)	Average potency (%)	Absorbance measured	RSD (%)	Average potency (%)
4	0.123	0.419	98.62	0.130	0.688	98.12
8	0.425	0.243	97.98	0.446	0.122	98.10
12	0.667	0.667	99.27	0.672	0.622	98.34

3.2.3 Stability

Stability study's results were within the acceptance range (Table 3) and indicated the samples stability over 24 h (short-term).

Table 3: Short term stability determined by the proposed method (n=3).

Concentration declared µg/mL	Concentration found µg/mL	RSD (%)	Average potency (%)
4	0.126	0.664	98.36
8	0.246	0.331	98.22
12	0.381	0.197	98.00

3.2.4 Accuracy/Recovery

Results within the range of 98.00–100.97% ensure an accurate method (Table 4) as well as indicate non-interference with the excipients of formulation.

Table 4: Accuracy/Recovery for three different concentrations of Tenoxicam by the proposed method

Dosage form	Label Claim	Amount added	Recovery (%)
Pre-formulated granules	200 mg	80	99.01
		100	99.25
		120	99.69

4. Conclusion

The results and the statistical parameters demonstrate that the proposed UV spectrophotometric method is simple, rapid, specific, accurate and precise. Therefore, this method can be used for the determination of Tenoxicam either in bulk or in the dosage formulations without interference with commonly used excipients and related substances.

5. Acknowledgment

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