RESEARCH PAPER

A Novel UV Spectroscopic Stability Indicating Method Development And Validation For The Estimation Of Oxaceprol API And Its Pharmaceutical Formulation

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Structured Abstract

A simple, sensitive, and selective UV Spectrophotometric method was successfully developed and validated for the estimation of OXACEPROL. The techniques utilized UV spectrometry, with the maximum absorbance (λmax) observed at 211 nm. The optimized concentration was determined to be 20 ng/ml, corresponding to an absorbance of 0.557. The assay method demonstrated excellent validation parameters, with a linearity coefficient of 0.9993. Precision and accuracy obtained a %RSD of less than 2, while recovery studies yielded results within the range of 98-102% (%RSD). Various stability studies, including LOQ, LOD,& robustness, confirmed the method's reliability, with LOD & LOQ values below 10. Robustness testing of % assay results ranging from 98-102%. Stability studies indicated the method remained stable under various conditions, and degradation studies under acidic and basic environments revealed less than 10% degradation, with % assay values maintained between 98-102%. The method was also effectively applied to the routine analysis of oxaceprol using UV spectrophotometry, where LOD and LOQ values were determined to be 1.21 ng/ml and 3.68 ng/ml.

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Introduction

Oxaceprol is a chondroprotective and anti-inflammatory agent widely used in the management of osteoarthritis and other degenerative joint disorders. Chemically, oxaceprol is the D-isomer of N-acetyl-L-proline and belongs to the class of amino acid derivatives. It exhibits unique anti-inflammatory activity by modulating leukocyte adhesion and migration rather than inhibiting cyclooxygenase (COX) enzymes, thereby reducing inflammatory responses without causing significant gastrointestinal adverse effects commonly associated with non-steroidal anti-inflammatory drugs (NSAIDs).Osteoarthritis is one of the most prevalent musculoskeletal disorders worldwide, characterized by progressive cartilage degeneration, joint pain, stiffness, and reduced mobility. With increasing life expectancy and aging populations, the global burden of osteoarthritis continues to rise. Oxaceprol plays a therapeutic role by improving joint function, reducing pain, and enhancing cartilage metabolism. Its mechanism of action involves inhibition of leukocyte rolling and adhesion to the endothelium, thus preventing inflammatory cell infiltration into synovial tissue. Oxaceprol is commonly available in oral dosage forms such as tablets and

capsules, typically in strengths of 200 mg and 400 mg. Due to its widespread clinical use, accurate and reliable quantification of oxaceprol in bulk drug and pharmaceutical formulations is essential to ensure product quality, safety, and therapeutic efficacy. Several analytical methods have been reported for the estimation of oxaceprol, including high-performance liquid chromatography (HPLC)

2. Chemical and Spectral Characteristics

The molecular structure of **Oxaceprol** contains:

- Carboxylic acid (–COOH) group
- Amide (peptide-like) linkage
- Heterocyclic ring system
- Secondary amine group
- Carbonyl (C=O) functional groups
- These structural elements act as chromophores responsible for UV absorption. The carbonyl (C=O) groups show **n → π*** electronic transitions, while the heterocyclic and conjugated systems contribute to **π → π*** transitions, resulting in absorption in the uv region. In acidic medium, protonation of the amine group enhances molecular stability and produces a characteristic absorption peak around **210–220 nm** in the UV spectrum.is shown in fig.1

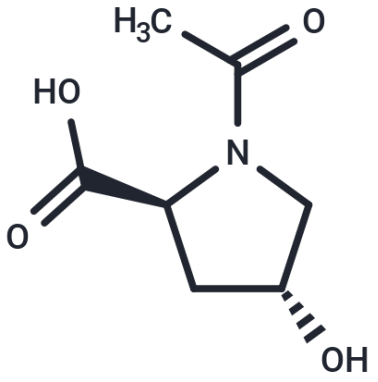


Fig.1. Structure of oxaceprol

3. Need for Analytical Method Development

Previously reported analytical techniques include:

- UV spectrophotometric methods
- RP-HPLC methods
- Stability-indicating chromatographic methods

While chromatographic methods offer higher sensitivity and separation capability, they require:

- Expensive instrumentation
- Organic solvents
- Skilled personnel
- Longer analysis time

For routine quality control laboratories, especially in developing countries, UV spectrophotometry offers:

- Rapid analysis
- Low operational cost
- Minimal solvent consumption
- Ease of method transfer

Therefore, development of a validated, stability-indicating UV method remains relevant and valuable.

Materials and Methods

3.1 Chemicals and Reagents

- Oxaceprol (API, working standard)
- Distilled water
- Concentrated hydrochloric acid
- DMSO
- Hydrogen peroxide (3%)

All reagents were of analytical grade and used without further purification

3.2 Instrumentation

All absorbance measurements were performed using a Shimadzu UV-1800 UV–Visible spectrophotometer equipped with matched 1 cm quartz cuvettes. The instrument was calibrated prior to analysis to ensure wavelength accuracy and photometric precision.

3.3 Determination of λmax

A 10 µg/mL standard solution of oxaceprol was scanned in the wavelength range of 200–400 nm using water as blank. The spectrum revealed a distinct absorption maximum at **211 nm**, corresponding to π→π* electronic transitions within the aromatic quinoxaline chromophore.

This wavelength was selected for further quantitative analysis due to optimal absorbance intensity and minimal baseline interference.

This wavelength was selected for quantitative estimation due to:

- Maximum sensitivity
- Sharp peak symmetry
- Minimal baseline interference

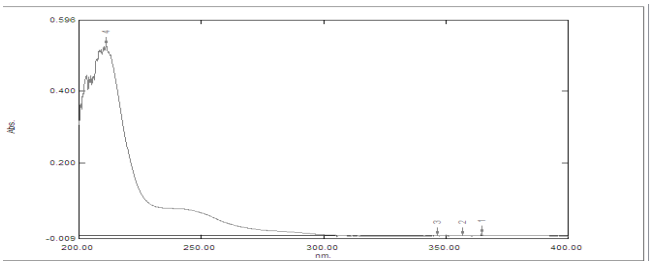


Fig.3. Selected wavelength

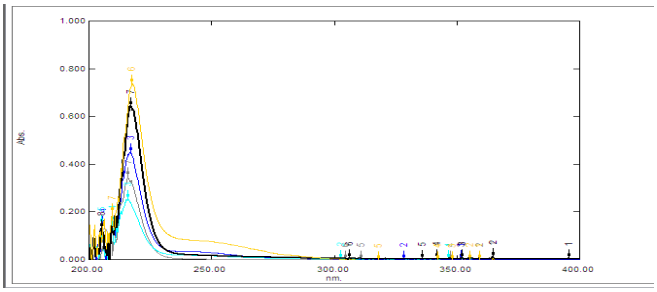


fig 4:Overlay of oxaceprol

3.4 Preparation of Standard Stock Solution

Weigh about 10 mg of oxaceprol API was dissolved in 50% DMSO to prepare the stock solution. From this 0.1 mL was diluted to 10 mL to obtain the primary stock solution (10 µg/mL). Further dilution of 0.1 mL to 10 mL gave the secondary stock solution (1 µg/mL). Pipette out 0.2 ml of the primary stock solution into a 10 ml volumetric flask and added distilled water to make the total volume. This gave us a 20 ng/ml solution

Preparation of Stock Solution

Accurately weigh 20 capsules and calculate the average weight of a single capsule. Separate the drug from the capsules Weigh out the equivalent of 11.875 mg of oxaceprol from the powdered capsule and transfer it into a 10 mL volumetric flask. Pipette out 0.2 ml from the above solution into a 10 ml volumetric flask and added distilled water to make the total volume. This gave us a 20 ng/ml solution.

Preparation of Working Standards

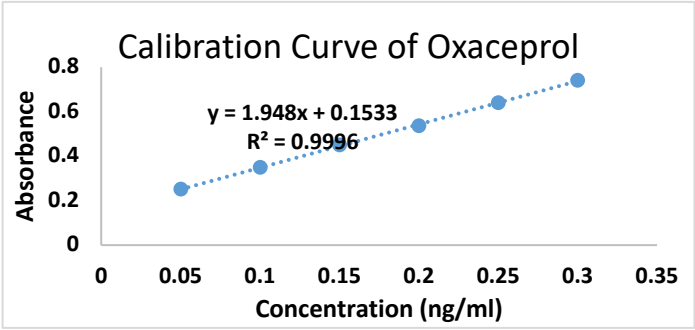
Aliquots of stock solution were appropriately diluted to obtain concentrations ranging from **5-30 ng/mL**.

3.5 Construction of Calibration Curve

Absorbance values were recorded at 211nm for each working standard. A calibration curve was plotted between concentration and absorbance.

The regression equation demonstrated linearity within the range 5–30 ng/mL with correlation coefficient (r^2) = **0.9993**, confirming adherence to Beer–Lambert's law.

Calibration curve was constructed (**Fig. 2**) and linear regression equation obtained.



4.Method Validation

Validation was performed in accordance with ICH Q2 (R1) guidelines.[12].

4.1 Linearity

The calibration curve was linear over 5–30 ng/mL (Table 1). $r^2 = 0.9996$.

The high correlation coefficient indicates proportional response in between concentration and absorbance.

The correlation coefficient confirms adherence to Beer–Lambert's law.

Table No.1: Linearity values of Oxaceprol

Concentration (ng/ml)	Absorbance
0.05	0.25
0.1	0.34
0.15	0.45
0.2	0.53
0.25	0.64
0.3	0.74

4.2 Accuracy

Accuracy was evaluated by recovery studies at 50%, 100%, and 120% spiking levels.

The mean percentage recovery ranged between **98-102%**, demonstrating excellent accuracy and without interference from excipients. (Table 2).

This confirms absence of interference and high trueness.

Table No.2: Showing Accuracy results

S.No	Spike Level	Amount taken (ng/ml)	Amount Added	Absorbance	Amount Recovered (ng/ml)	(%) Recovery	Mean (%) Recovery

			(ng/ml)				
1	50%	0.2	0.3	0.725	0.302	101.27	101.36
2			0.3	0.73	0.304	101.96	
3			0.3	0.722	0.300	100.85	
4	100 %	0.2	0.4	0.962	0.400	100.10	100.45
5			0.4	0.954	0.399	99.79	
6			0.4	0.97	0.406	101.46	
7	150 %	0.2	0.5	1.19	0.495	99.73	99.94
8			0.5	1.18	0.494	99.41	
9			0.5	1.195	0.500	100.67	

4.3 Precision

Precision was assessed through intra-day and inter-day studies.

- **Intra-day precision**: %RSD = 1.51
- **Inter-day precision**: %RSD = 1.8

Both values were within acceptable ICH limits (<2%), confirming method reproducibility.
(Tables 3 & 4)

TableNo.3 Inter-Day Precision Performance of the Proposed Method

S.NO	INTERDAY PRECISION (ABSORBANCE)		
	Day 1	Day 2	Day 3
1	0.553	0.505	0.536
2	0.546	0.522	0.543
3	0.548	0.52	0.556
4	0.557	0.516	0.536
5	0.563	0.528	0.535
6	0.551	0.518	0.538
Mean	0.007767	0.007	0.009195
STD	1.409701	1.351351	1.707069
%RSD	1.74	1.86	1.45

Table No.4 : Intra-Day Precision Performance of the Proposed Method

S.NO	INTRADAY PRECISION(ABSORBANCE)
1	0.549
2	0.547
3	0.552
4	0.557
5	0.568
6	0.576
Mean	0.558167
STD	0.010511
%RSD	1.60

4.4 Limit od detection and limit of quantification

Calculated using formula:
LOD = 3.3σ/S
LOQ = 10σ/S
Values obtained were 0.018 ng/mL and 0.05 ng/mL (Table 5). These values demonstrate adequate sensitivity.

Table No.5: LOD and LOQ Values Obtained from Calibration Curve

S.NO	Parameter	Standard Deviation	Slope	LOD&LOQ(ng/ml)
1	Limit of detection (LOD)	0.0105	1.948	0.018
2	Limit of quantitation (LOQ)			0.05

4.5 Robustness

Robustness was evaluated by introducing small deliberate variations in wavelength (±3 nm) & Minor solvent changes The percentage %RSD values were below 2%, demonstrating that minor variations did not significantly affect analytical performance. (Tables 6 & 7). Indicates reliability under small variations.

Table No.6&7: Results showing Robustness data

S. No	Change in Parameter		Absorbance			Mean	Std. Dev	% RSD
1	Solvent Proportion (± 5%)	45	0.552	0.573	0.569	0.564	0.0091043	1.61
2		50	0.553	0.546	0.548	0.549	0.0029439	0.53
3		55	0.568	0.579	0.574	0.573	0.0044969	0.78

4	Wavelength (± 3 nm)	208	0.517	0.505	0.518	0.513	0.005906	1.15
5		211	0.539	0.553	0.546	0.546	0.005715	1.04
6		214	0.522	0.52	0.516	0.519	0.002494	0.48

4.6 Forced Degradation Studies

Stress studies were conducted under acidic, alkaline, oxidative, thermal, and photolytic conditions. Minimal degradation was observed (Table 8). Overlay spectrum shown in **Fig. 5**. Maximum degradation observed in oxidative condition (0.78%).

Table No.8: Stress Degradation studies of Oxaceprol

S.No	Stress conditions	Absorbance	% Assay	% Degradation
1.	0.1M HCL	0.551	97.6	2.4
2.	0.1M NaOH	0.514	104.6	2.4
3.	Hydrogen peroxide (H2O2) 3% v/v	0.542	99.22	0.78
4.	Thermal (70ºC)	0.529	101.66	5.82
5.	Photolytic (UV)	0.571	94.18	1.69

CONCLUSION

A reliable, precise, and cost-effective UV spectroscopic method has been successfully developed for the estimation of oxaceprol in both bulk and in-house tablet formulations. This method is simple, rapid, and robust; making it suitable for routine analysis. The precision of the method is confirmed by a relative standard deviation (RSD) of less than 2%, indicating high reproducibility. Accuracy studies show a recovery rate between 98-102%, demonstrating excellent accuracy and specificity. Additionally, the high percentage of drug recovery confirms that excipients in the tablet formulation do not interfere with the analysis, proving the method's specificity and effectiveness. Given its reliability, the developed UV spectroscopic method is well-suited for the routine quality control analysis of oxaceprol in pharmaceutical formulations.

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