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New Visible Spectrophotometric Method Development for the Quantification of Lasmiditan in Bulk and Its Tablet Formulation

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Abstract: A reliable, precise & less time-consuming spectrophotometric method for Lasmiditan estimation by using chromogenic chelation approach is reported in this work. Method A uses 1, 10-Phenanthroline which involves redox and chelation reactions forming an orange-red Fe^{2+} complex with a λ-max at 530 nm. Method B uses 2, 2′-Bipyridine to form a stable orange-red Fe^{2+} complex with λ-max at 516 nm. Both obeys Beer–Lambert's law within 1-10 µg/mL range. The Method is validated & Sandell's sensitivity & other parameters was found to be within the regulatory limits.

Keywords: *Lasmiditan, 1,10-Phenanthroline, 2,2'-Bipyridine, Spectrophotometry, Quantitative estimation.*

INTRODUCTION:

Lasmiditan is a pyridinyl-piperidine derivative that belongs to the Ditan class[1] of anti-migraine agents which acts primarily through inhibition of trigeminal nerve activity and neuropeptide release, without inducing vasoconstriction making it safer for patients with cardiovascular risk factors.[3,4]

Few methods were reported[5,6] for its quantitative estimation, these methods are limited by high operational costs, complex sample preparation, and longer analytical times.[7,8] One Visible method with MBTH was found in the literature but the high cost of MBTH restricts its routine application in resource-limited settings and industrial quality control environments.[9]

To address this, the present study focuses on the development and comparison of two cost-effective visible spectrophotometric methods utilizing ferrous ions from 0.1M $FeSO_4 \cdot 7H_2O$ with two different chromogenic ligands. Method A employs 1,10-Phenanthroline forms a stable-colored complex, while Method B uses 2,2′-Bipyridine as a reagent, with absorbance observed at 516 nm.[9] The study is comparative in nature, and while both methods show measurable responses, Method A demonstrates better adherence to Beer–Lambert's law and superior analytical performance. These findings support industrial applications, offering a precise, reproducible, and economically viable alternative for Lasmiditan estimation in pharmaceutical formulations and in bulk drug.

DRUG PROFILE:

Lasmiditan

Lasmiditan is a benzamide derivative and the structure is shown below.

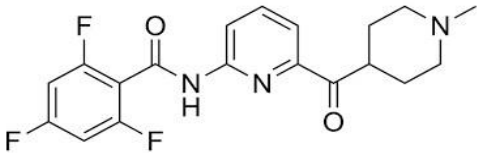

Fig 1: Lasmiditan structure.[9]

It contains a unique pyridinyl-piperidine scaffold contributing to its lipophilic nature and enhanced blood–brain barrier penetration.[1,2,3,4] The tablets containing excipients do not interfere in the estimation of API.[5,7,8]

Materials and Method:

Instruments

A Shimadzu UV-1700 with 1cm path length were used.[10,11] A Digisun pH meter, Shimadzu weighing balance was used for this Research.[12]

Reagents Used

Lasmiditan drug (API) was received from Pure and Cure Pvt. Ltd, Uttarakhand. 1,10-Phenanthroline from Sisco Pvt. Ltd. 2,2‘-Bipyridine AR was obtained froms.d.fine-Chem Limited, Mumbai 400025. Ferrous sulphate Heptahydrate was received from oxford lab fine chem llp. Ethanol AR was obtained from bio liqua research Pvt. Ltd. and is used for preparation of solutions along with double distilled water. Migditan™ 100 mg formulation was obtained from local market.

Stock Solution

1000 µg/mL (Stock-I) and 100 µg/mL (Stock-II) were prepared by using ethanol and Lasmiditan API.[7]

Working standard solution

Aliquotsof stock-II containing 0.1-1.0 mL of Lasmiditan used as working standard solution.[9]

Preparation of Reagents

A 0.25% w/v solution of 1,10-phenanthroline monohydrate was prepared by taking 0.25 gm of the compound in 100 mL of double distilled water in a beaker.[9] A 0.2% w/v solution of ferrous sulphate heptahydrate was obtained by dissolving 2.78 gm in 100 mL of water, followed by sonication for 10 min to ensure complete dissolution.[9] The sodium acetate buffer of pH 4.5 was made by dissolving 0.299 gm of sodium acetate in 0.166 mL of acetic acid and make up the final volume to 100 mL with distilled water.[9,11] Similarly, a 0.2% w/v solution of 2,2′-Bipyridine was obtained by taking 0.2 gm of the compound in 100 mL of distilled water.

Determination of λmax

A standard solution of Lasmiditan having 1,10-Phenanthroline & 2,2′-Bipyridine reagents was scanned in the visible region. The drug exhibited a well-defined absorbance peak at 530 nm & 516 nm respectively.[9,10,11]

ASSAY PROCEDURE FOR METHOD-A

Method for Bulk Drug estimation

A series of dilution of stock-II having 0.1-1.0 mL of Lasmiditan were taken into10 mL volumetric flasks, One mL of 1,10-Phenanthroline (0.25%w/v), 1.0mL of Ferrous Sulphate Heptahydrate (0.2%w/v) and OnemL of Sodium Acetate were added to each flask resulting in the formation of orange-red Fe^{2+} complex and make up the volume to 10mL with solvent ethanol. The absorbance is taken at 530nm. Bulk sample estimation was done by substituting sample absorbance values to get unknown concentration in regression equation.

Assay method for Formulation

Weigh and powder twenty tablets, take 100 mg equivalent weight of the powder & extract the Lasmiditan in Ethanol. The filtrate and washings were combined, and the final volume was made to 100 mL with ethanol to prepare Stock-I. Suitable sample is prepared within the range of Beer-Lambert limit and analyzed.

ASSAY PROCEDURE FOR METHOD-B

Method for Bulk Drug estimation

Aliquots of standard solutions of Stock-II containing 0.1–1.0 millilitre were taken in 10 mL flasks. To each of the flask, one mL of 2,2′-Bipyridine (0.2% w/v), 1.0 mL of Ferrous Sulphate Heptahydrate (0.2% w/v) & one millilitre Sodium Acetate were added to form a stable orange-red Fe^{2+} complex. The final volume was adjusted to 10 mL using ethanol and measurethe absorbance at 516 nm using blank prepared under identical conditions.[9,10] The unknown concentration of Lasmiditan in each bulk sample was determined from regression line.

Assay method for Formulation

Formulations were analyzed by extracting the sample similar to Method-A and 2,2′-Bipyridine was added as mentioned in the bulk drug estimation procedure. Suitable sample is prepared within the range of Beer-Lambert limit and analyzed.

Reaction complex of Lasmiditan with Iron and 1,10-Phenanthroline

1,10-Phenanthroline involves redox and chelation reactions forming an orange-red Fe^{2+} complex in presence of Lasmiditan. Proposed complex is shown in the (figure 2).

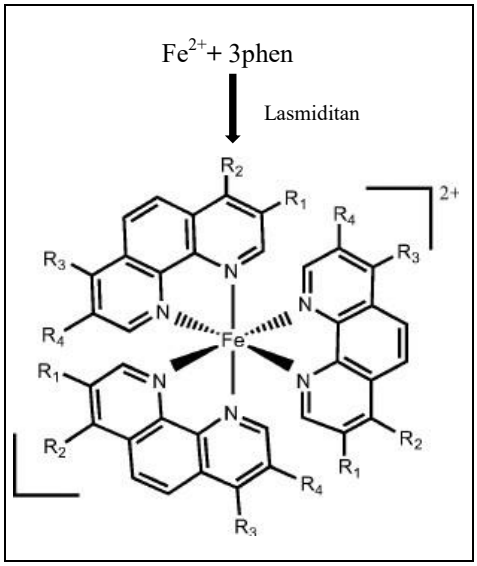

Fig 2: Ligand Complex formation with 1,10-Phenanthroline and Iron.[13]

Reaction complex of Lasmiditan with Iron and 2,2′-Bipyridine

2,2′-Bipyridine involves redox and chelation reactions forming an orange-red Fe^{2+} complex in presence of Lasmiditan. Proposed complex is shown in the (figure 3).

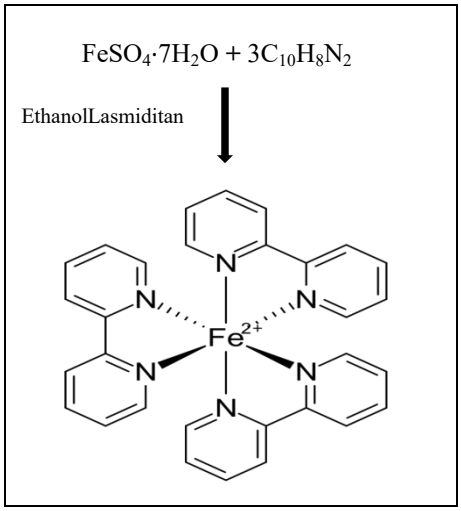

Fig 3: Ligand Complex formation with 2,2-Bipyridine and Iron.[14]

Results & Discussion:

Table 1: λ-max estimation for Method-A

Wavelength (nm)	Absorbance
520	0.043
522	0.088
524	0.101
526	0.112
528	0.147
530	0.241
532	0.198
534	0.112
536	0.073
538	0.051
540	0.030

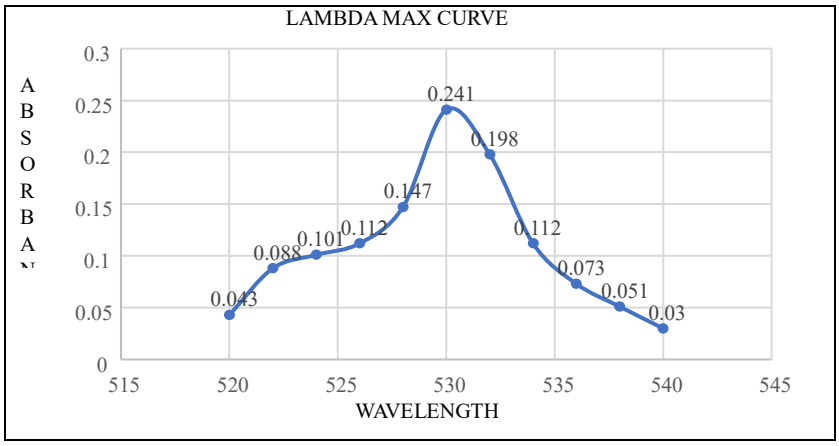

Fig 4: Absorption spectrum of Lasmiditan for Method-A.

Table 2: Linearity plot values for Method-A

Conc (µg/mL)	Absorb
1	0.241
2	0.312
3	0.432
4	0.523
5	0.587
6	0.689
7	0.780
8	0.870
9	0.960
10	1.051

Table 3: Optical characteristics for Method-A

Sl.No	Parameter	Description/Result
01	Analytical method	Visible Spectrophotometric
02	λ_{max} (nm)	530
03	Beer's range	1-10µg/ml
04	Regression line	y = 0.0902x + 0.1482
05	Regression coefficient (r^2)	0.9988
06	SD	0.926
07	SEM	0.535
08	Sandell's Sensitivity	0.011
09	Formulation Analyzed	Lasmiditan (MIGDITAN-100MG)
10	Percentage Recovery	99.05

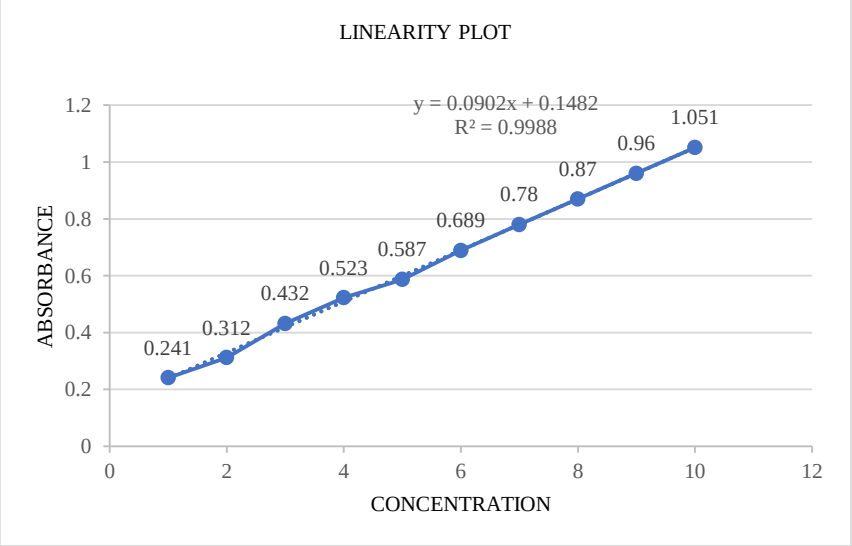

Fig 5: Linearity plot for Method-A.

Table 4: .λ-maxfor Method-B

Wavelength (nm)	Absorbance
506	0.058
508	0.111
510	0.143
512	0.177
514	0.209
516	0.318
518	0.236
520	0.185
522	0.157
524	0.101
526	0.069

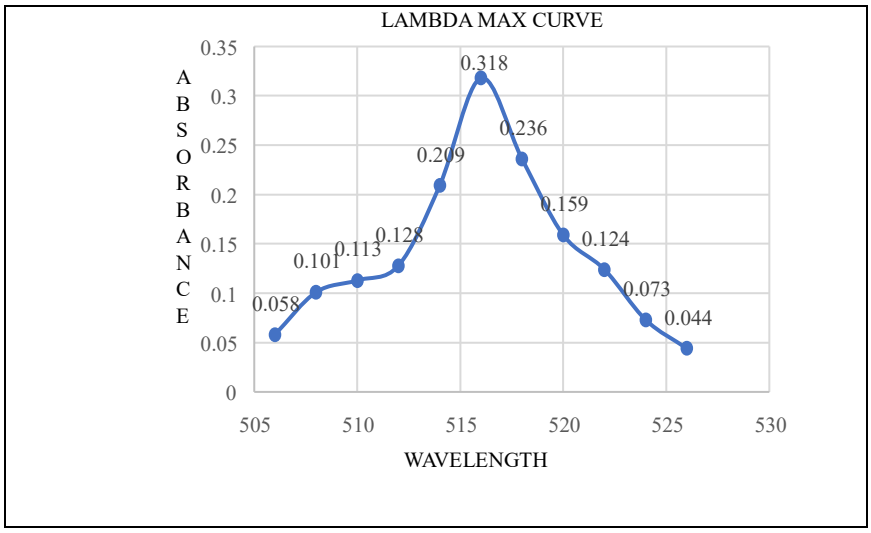

Fig 6: Absorption spectrum of Lasmiditan for Method-B.

Table 5: Optical characteristics for Method-B

Sl.No	Parameter	Description/Result
01	Analytical method	Visible Spectrophotometric
02	λ_{max} (nm)	516
03	Beer's	1-10µg/ml
04	Regression line	y = 0.0902x + 0.1482
05	Regression coefficient (r^2)	0.9988
06	Standard Deviation	1.520
07	Standard Error of Mean	0.878
08	Sandell's Sensitivity	0.121
09	Formulation	MIGDITAN
10	% Recovery	98.032

Table 6: Linearity plot values for Method-B

Conc (µg/mL)	Absorbance
1	0.381

2	0.531
3	0.856
4	0.903
5	0.992
6	1.166
7	1.322
8	1.477
9	1.6324
10	1.7876

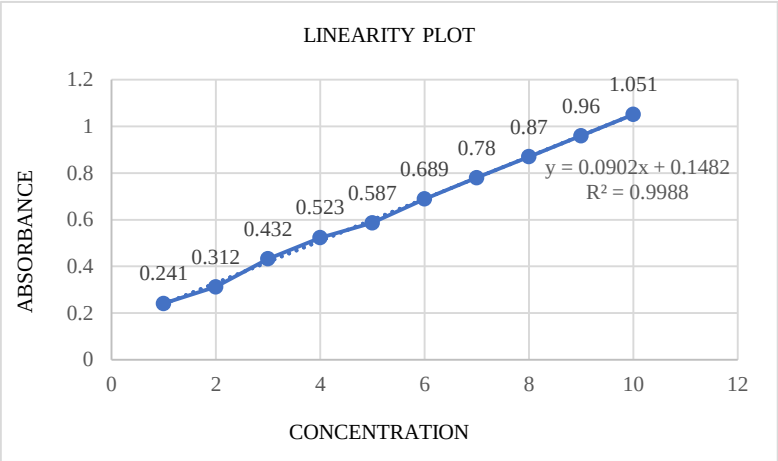

Fig 7: Linearity plot for Method-B.

Linearity and Calibration

A comparative linearity and calibration studies were performed for both Methods. Method A exhibited a well-defined and consistent linear relationship between absorbance and concentration across the tested range, demonstrating strong adherence to Beer–Lambert's law. The absorbance readings were stable and showed minimal variability, confirming the reliability.

Accuracy

It is determined by how closely the measured value matches the sample's actual value, three distinct drug concentration levels 80%, 100%, and 120% were made from standard analytes examined.[23] The mean percentage recovery and relative error percentages were used to measure accuracy for both methods.

Table 7: MethodA- Accuracy

Drug added RSD %	Lasmiditan, μgmL^{-1}	Lasmiditan (μgmL^{-1})	Added, (μgmL^{-1})	Found (μgmL^{-1})	% Recovery	Average	% RSD
80	5	4.6	9.6	9.6 9.4 9.2	100 98.14 96.2	9.4	1.94%
100	5	5	10	10 9.8 6.1	100 98.33 101.66	10	1.67%
120	5	5.4	10.4	10.4 10.3 10.4	100 99.08 100	10.36	0.533%

Table 8:.Accuracy of Method-B

Drug added RSD %	Lasmiditan, μgmL^{-1}	Lasmiditan (μgmL^{-1})	Added, (μgmL^{-1})	Found (μgmL^{-1})	% Recovery	Average	% RSD
80	10	8.8	18.8	18.8 18.4 18.9	100	18.7	1.41%
100	10	10	20	20 20 20.2	100	20.06	0.575%
120	10	12.2	22.2	22.2 22.3 22.4	100	22.3	0.448%

Precision

Lasmiditan concentrations that were similar to those used in the accuracy research were employed to evaluate the repeatability of the proposed procedures. To guarantee procedure reliability, these concentrations were made from standard analyte solutions and examined. Inter-day and intra-day variability experiments were conducted. [23] Six replicates with varied concentrations were made and examined three times in a single day to ensure intra-day precision. To investigate inter-day precision, the identical process was carried out three days in a row. One set of prepared concentrations was reanalyzed using spectrophotometer in order to evaluate inter-instrument precision. Precision was measured using the concentration's percent relative standard deviation (% RSD), which was derived using the regression equations. Precision values are illustrated in following table.

Table 9: Precision data

Intra-dayInter-day

Found (µg/mL)		Average		%RSD		Found(µg/mL)		Average		%RSD	
A	B	A	B	A	B	A	B	A	B	A	B
4	8	4.124	8.02	0.368%	3.78%	4	8	4.412	8.4	0.495%	3.47%
4.11	8.2					4.14	8.4				
4.14	7.8					4.16	8.8				
4.14	7.6					4.14	8.5				
4.11	8.2					4.16	8.0				
4.12	8.3					4.11	8.3				

Sandell's sensitivity (SS)

The amount of substance (in micrograms) that produces an absorbance of 0.001 in a 1 cm path length cuvette at a specified wavelength. It is a useful parameter in UV-Visible spectrophotometry to assess how small an amount of analyte can be detected visually by a developed method.[24]

$$\text{Sandell's Sensitivity (SS)} = \frac{0.001}{\text{slope of the calibration curve (Abs / } \mu g/cm^2)}$$

Recovery Studies

Recovery studies in spectroscopic method development were conducted to evaluate method accuracy by determining the percentage of analyte recovered from a sample matrix after spiking with known concentrations.[15,19] A suitable blank or real matrix is selected, and standard analyte solutions are prepared at 80%, 100%, and 120% of the target concentration. Known amounts of these standards are added to the matrix, and both spiked and unspiked samples are analyzed in triplicate using the developed method. Recovery is calculated using the formula:

$$\text{Recovery (\%)} = \left[\frac{\text{Amount found after spiking} - \text{Amount in unspiked sample} \times 100}{\text{Amount Added}}\right]$$

Table 10: Recovery studies

Method	Label Claim (mg)	% Label Claim Found	Spiking%	Standard Deviation	Coefficient of Variation	Standard Error	% Recovery
Method-A (1,10-Phenanthroline)	100	101	80.43	0.926	1.152	0.535	99.701
Method-B (1,2'-Bipyridine)	100	99.9	78.3	1.520	1.941	0.878	98.032

DISCUSSION:

Few Liquid Chromatographic and one Spectroscopicmethods were reportedin the literature but the proposed methods (A & B) are economic, less-time consuming, and effective in routine analysis. The wave maxima of method-A was found to be 530 nmand this wavelength is independent of concentration. In this study 1,10-Phenanthroline and Iron were used to form chromogenic complex with Lasmiditan. Similarly, the λ-max for Method B was found to be 516 nm, where the chelate complex is formed by the reaction of 2,2′-bipyridine with Iron. The proposed methods were linear obeying Beer's Law. Average recoveries werein between 98% - 101%.

CONCLUSION:

The proposed methods are sensitive, reliable & they do not require any critical extraction or complex sample preparation, and are unaffected by the presence of common excipients and other active ingredients in pharmaceutical formulations. The developed methods have been validated through various analytical parameters including accuracy, precision and Sandell's Sensitivity. The results confirm the reproducibility of the methods, demonstrating their suitability.

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