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Original Article

NEW DIFFUSE REFLECTANCE INFRARED FOURIER TRANSFORM SPECTROSCOPY FOR THE ESTIMATION OF TRAMADOL HYDROCHLORIDE CAPSULES

VENKATA SAI GANESHA PRAVALLIKA BURELA, USHA RANI NAMMI, PRANATI MANDALEMULA

Maharajah's College of Pharmacy, Vizianagaram, India
Email: pravallikapravi1713@gmail.com

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ABSTRACT

Objective: The objective of the study was to develop and validate a new diffuse reflectance infrared fourier transform (DRIFT) spectroscopic method for the quantification of Tramadol HCl in the pharmaceutical dosage form.

Methods: The Fourier Transform Infrared (FTIR) analysis was carried out on Cary 630 FTIR spectrophotometer (Agilent Technologies, USA) equipped with a diffuse reflectance sampling interface (650-4000 cm^{-1}, 32 scans, 8 cm^{-1} resolution). The solid-state samples were prepared by dilution in dry potassium bromide and were analysed by an FTIR spectrophotometer with the DRIFT sampling technique, which follows the Kubelka-Munk model depicting the theory of diffuse reflectance at scattering surfaces, relating band intensities to concentration for transmission measurements similar to Beer's law.

Results: A linear relationship was found in the selected wavenumber range of 3295-3298 cm^{-1}denoting the carboxyl peak in the concentration range of 0.6-3.0% w/w with a good correlation coefficient of 0.997. The percent recovery of Tramadol HCl in the marketed dosage form was found to be 100.4%.

Conclusion: The proposed method was found to be accurate, precise, reproducible, and eco-friendly. DRIFT spectroscopy may have the potential as an alternative method for qualitative and quantitative analysis of Tramadol HCl in its capsules.

Keywords: Tramadol hydrochloride, Diffuse reflectance infrared fourier transform spectroscopy (DRIFTS), Validation, Quantification

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INTRODUCTION

Tramadol HCl is chemically (+)-cis-2-[(dimethylamino) methyl]-1-(3-methoxy-phenyl) cyclohexanol hydrochloride belongs to the class of drugs known as opioid analgesics [1-3]. It is a centrally-acting opioid agonist and serotonin/norepinephrine reuptake inhibitor used for the management of moderate to severe pain in adults [4]. The extended-release capsules and tablets are used for chronic ongoing pain.

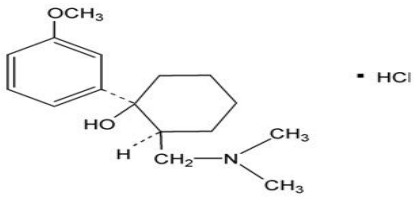

Fig. 1: Structure of tramadol hydrochloride

DRIFT spectroscopy is a relatively newer IR technique that has shown potential for the quantification of solid samples. DRIFT spectroscopic methodology has the advantages of being the least complex procedures involving simple sample preparation, eco-friendly to analyse solid-state pharmaceuticals and do not need the drug extraction procedures [5]. The use of DRIFT spectroscopy for the identification of pharmaceuticals almost eliminated the use of classical identification techniques such as color and chemical tests.

DRIFT Spectroscopy allows for collecting spectra from powdered samples [6]. The practical difficulty that may arise for the quantification of solid-state samples using ATR and transmission measurement is, lack of reproducibility, which can be minimised by the proposed DRIFT sampling technique used in the present study [7].

The literature survey revealed that many analytical methods like Colorimetry [8], UV spectrophotometry [9], HPTLC [10] and HPLC [11-13] were reported for the estimation of Tramadol HCl in pharmaceutical formulations, which involved lengthy sample preparations using expensive solvents. To the best of our knowledge, there is no published literature data on a solvent-free analytical method for the quantitation of Tramadol HCl in pharmaceutical dosage form using FTIR. Hence, the authors made an attempt to develop and validate a new solvent-free FTIR method for quantification of Tramadol HCl in solid-state and evaluate the feasibility of DRIFT application for analysis of pharmaceuticals.

MATERIALS AND METHODS

Reagents

A standard sample of Tramadol HCl was obtained from Arochem Laboratories Private Ltd., Mumbai. Potassium bromide (Analytical Grade) was obtained from Lotus Enterprises Private Ltd., Mumbai.

Instrument

All the diffuse reflectance spectra were collected using a Cary 630 FTIR spectrophotometer (Agilent Technologies, USA) equipped with a diffuse reflectance sampling interface. FTIR spectra were recorded in the wave number range between 4000 and 650 cm^{-1}, averaging 32 scans per sample using a nominal resolution of 8 cm^{-1}. Cary 630 Micro Lab PC Software was used for data acquisition and analysis.

DRIFT spectroscopic method

In the proposed DRIFT technique, the IR beam is reflected and dispersed on the matt surface of the sample. The emerging radiation

leaves the sample in all directions and is directed towards the detector. In this study, we report a validated DRIFT spectroscopic method for quantitative analysis of Tramadol HCl in solid dosage forms. Appropriate quantities of Tramadol HCl and dry potassium bromide were mixed to get the desired working standards for spectral analysis using a diffuse reflectance sampling interface [6, 7]. The peak in the fourier transform infrared spectrum around 3295-3298 cm^{-1} was observed for the carboxyl group of Tramadol HCl. A linear calibration curve with a correlation coefficient of 0.997 was obtained in the range of 0.6-3.0% w/w of Tramadol HCl. This method was validated as per ICH guidelines and then applied to commercial formulations.

RESULTS AND DISCUSSION

Analytical wave number selection

Tramadol HCl was scanned in the wavenumber range of 650-4000 cm^{-1} with resolution of 8 and 32 scans. The functional group selected for Tramadol HCl was the carboxyl group, and the wave number found was in the range of 3295-3298 cm^{-1}. The reflectance and absorbance spectra for the standard Tramadol HCl were shown in fig. 2-3 [14]. Reflectance spectra of corresponding working standards were shown in fig. 4-8, respectively.

Validation parameters

The developed DRIFT spectroscopic method was validated for linearity, accuracy and precision as per ICH guidelines [15].

Linearity

The linearity of the proposed method was studied by analysing three replicates of each working standard (0.6-3.0% w/w) of Tramadol HCl. The regression coefficient of the calibration curve as shown in fig. 9 was found to be 0.997, indicating good linearity (table 1).

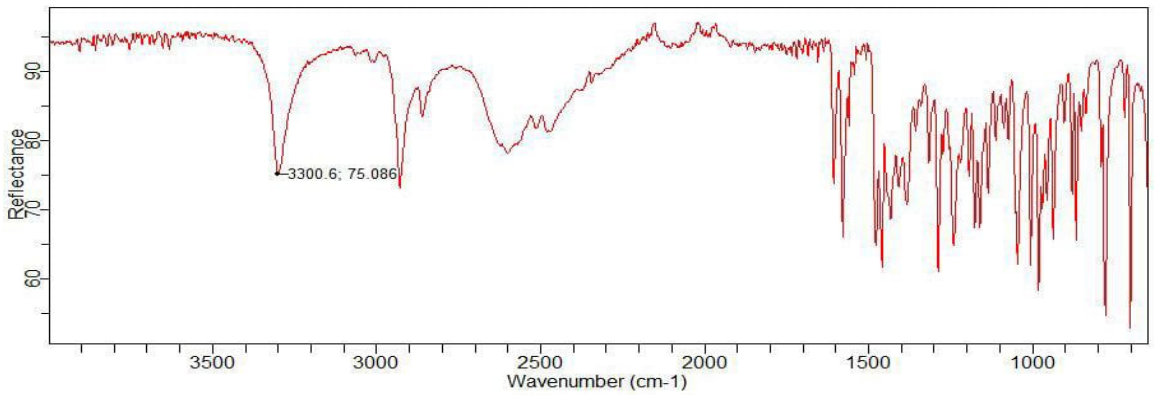

Fig. 2: Reflectance spectrum of standard tramadol hydrochloride

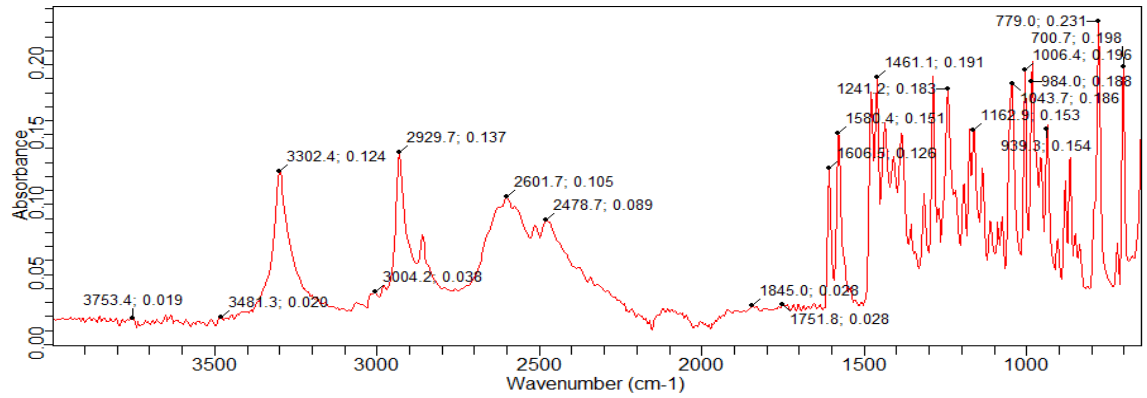

Fig. 3: Absorption spectrum of standard tramadol hydrochloride

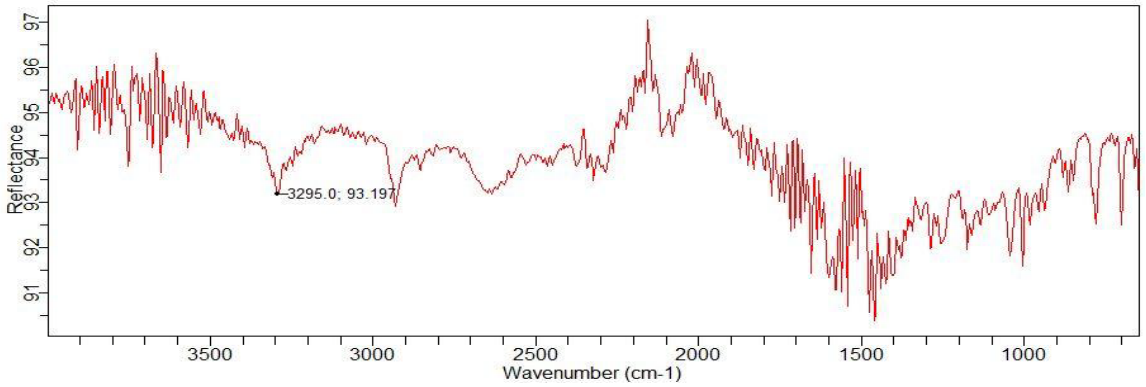

Fig. 4: Reflectance spectrum of tramadol hydrochloride sample 0.6% w/w in potassium bromide

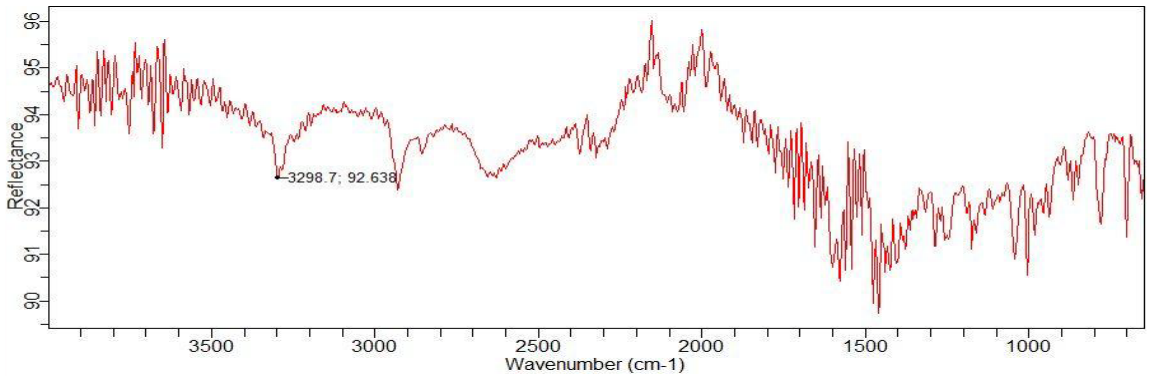

Fig. 5: Reflectance spectrum of tramadol hydrochloride sample 1.2% w/w in potassium bromide

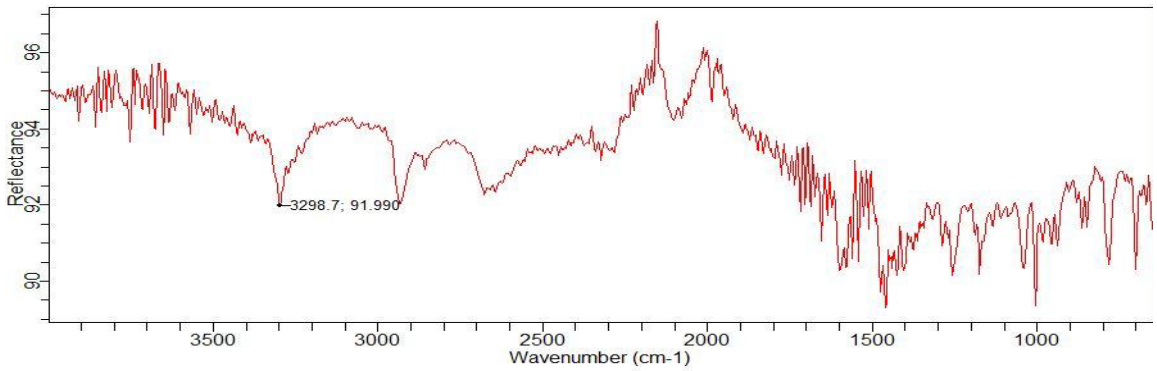

Fig. 6: Reflectance spectrum of tramadol hydrochloride sample 1.8% w/w in potassium bromide

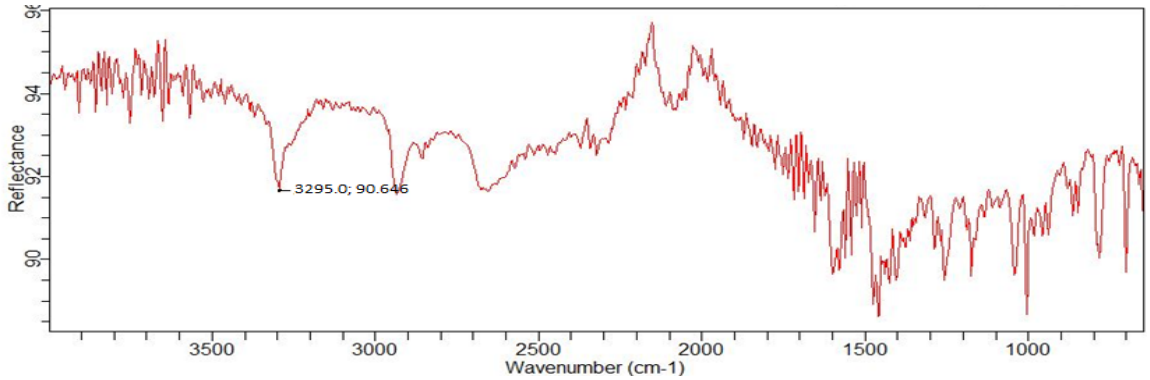

Fig. 7: Reflectance spectrum of tramadol hydrochloride sample 2.4% w/w in potassium bromide

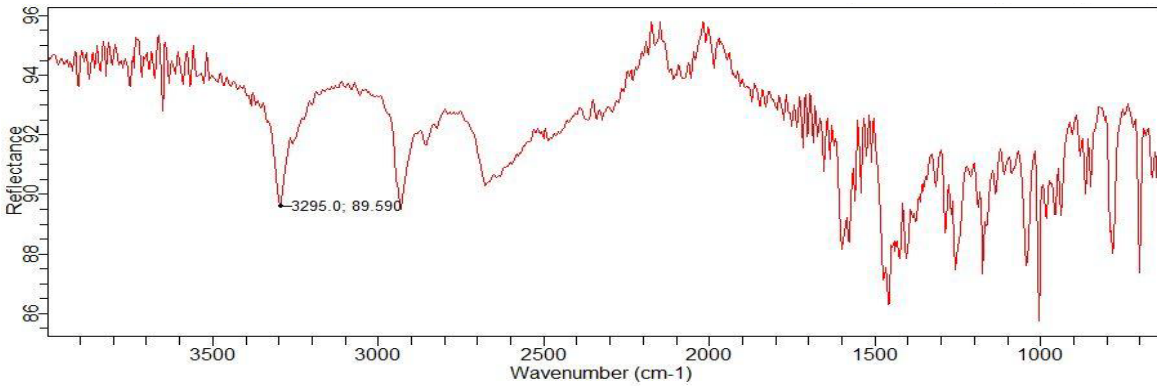

Fig. 8: Reflectance spectrum of tramadol hydrochloride sample 3.0% w/w in potassium bromide

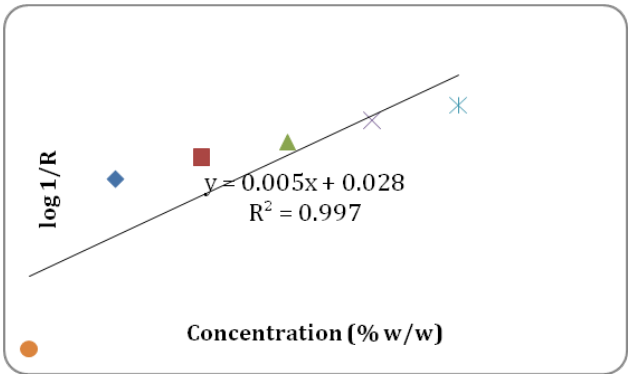

Fig. 9: Calibration curve of tramadol hydrochloride (0.6-3.0% w/w)

Table 1: Linear regression data of the proposed method

S. No.	Statistical parameters	Values
1	Concentration range	0.6-3.0% w/w
2	Regression equation	y = 0.005x+0.028
3	Correlation coefficient	0.997

Accuracy

The accuracy of the proposed method was evaluated by standard addition procedure. To the preanalyzed Jagdol-x capsule powder, a known amount of Tramadol HCl standard powder corresponding to 80, 100 and 120% of the label claim was added and analyzed. The recovery data is shown in table 2.

Precision

Precision of the method was assessed by repeatability and intermediate precision studies. Repeatability studies were performed by analysing samples of three different concentrations (1.2-2.4% w/w) of Tramadol HCl thrice on the same day (day 1). The intermediate precision was evaluated by interday studies (three consecutive days). The results are shown in tables 3 and 4, respectively.

Analysis of marketed formulation

The applicability of the proposed DRIFT technique on the commercial formulation (Jagdol-x) was accessed. Twenty capsules were taken, the contents pooled, and the average weight was found. An appropriate quantity of powder equivalent to 50 mg of Tramadol HCl was mixed with KBr powder, further diluted to 1% w/w concentration and analysed in the range of 3295-3298 cm^{-1} using KBr as blank. The assay results are shown in table 5, and the corresponding FTIR spectrum is shown in fig. 10.

Table 2: Recovery data of the proposed method

Brand name	Label claim (mg)	Amount of drug added (mg)	Amount of drug recovered* (mg) mean±SD (n=3)	Recovery (%)
Jagdol-x	50	40	90.37±0.45	100.4
	50	50	100.4±0.30	100.4
	50	60	110.4±0.36	100.3

*Average of three determinations

Table 3: Intra-day precision data of the proposed method

S. No.	Concentration(% w/w)	Reflectance* mean±SD (n=3)	% RSD
1	1.2	92.329±0.45	0.48
2	1.8	91.718±0.52	0.56
3	2.4	90.548±0.22	0.24

*Average of three determinations

Table 4: Inter-day precision data of the proposed method

S. No.	Concentration (% w/w)	Reflectance*			mean±SD (n=3)	% RSD
		Day 1D 2 D3				
1	1.2	92.646	92.412	92.096	92.384±0.27	0.29
2	1.8	91.842	91.268	91.045	91.385±0.40	0.44
3	2.4	90.686	90.358	90.548	90.530±0.15	0.16

*Average of three determinations

Table 5: Assay results

S. No.	Brand name	Absorbance*	Label claim (mg)	Amount recovered (mg)	Recovery (%)
1	Jagdol-x	0.042	50	50.2	100.4

*Average of three determinations

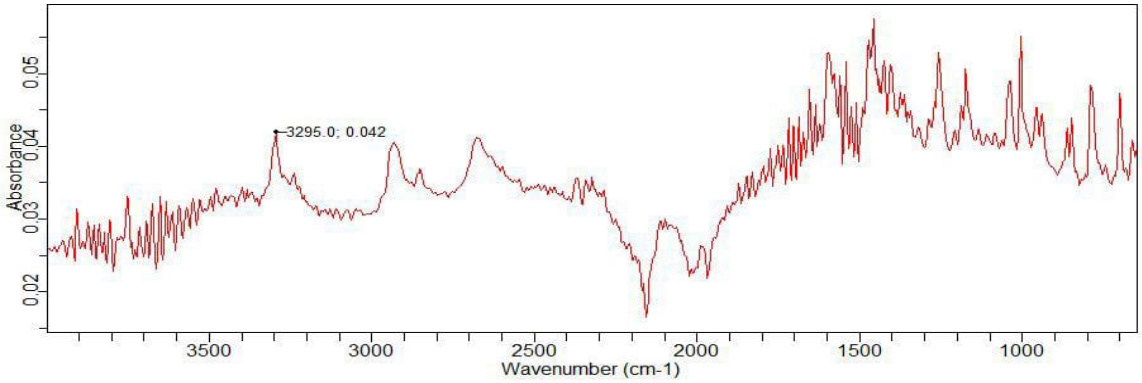

Fig. 10: FTIR spectrum for the marketed formulation diluted with potassium bromide

DRIFT spectroscopy is typically applied to analyse powders and rough surface solids. This sampling technique is used on powder samples, involving simple sample preparation. Since organic compounds exhibit intense absorption peaks in the 4000-650 cm^{-1}region of the IR spectrum, it is possible to quantitatively analyse them using DRIFT spectroscopy. The reflectance spectrum of standard Tramadol HCl was shown in fig. 2.

The FTIR spectrum for a pure sample of Tramadol HCl exhibited absorbance bands at 3753, 3481, 3302, 3004, 2929, 2601, 2478, 1845, 1751, 1606, 1580, 1461, 1241, 1162, 1043, 1006, 984, 939, 779 and 700 cm^{-1} as shown in fig. 3. The reflectance spectra corresponding to the carboxyl group were found in the range of 3295-3298 cm^{-1} for working standards of Tramadol HCl in potassium bromide as shown in fig. 4-8. The log 1/R value corresponding to the carboxyl peak around 3295-3298 cm^{-1} was used for the quantification. The calibration curve was constructed by plotting the log 1/R value versus nominal concentrations (0.6-3.0% w/w) and the regression equation was found to be "y=0.005x+0.028" with a correlation coefficient of 0.997, indicating good linearity (table 1).

The accuracy of the proposed method was evaluated by the recovery of pure drug from excipients at three different concentration levels (80, 100 and 120% w/w of label claim) by the standard addition method. Good recovery of Tramadol HCl was found in the range of 100.3-100.4%, showing that the method is quite accurate (table 2). The results of recovery studies supported that there was no interference of excipients in the analysis. Hence, the proposed method may have the potential to serve for in-house quality control of Tramadol HCl. The % RSD values for intraday precision studies and interday precision studies were found to be 0.24-0.56% and 0.16-0.44%, respectively, indicating that the method is quite precise (table 3 and 4). The present validated method was applied for the quantification of Tramadol HCl in capsule dosage form, where the recovery was found to be 100.4% (table 5), indicating that the method can be successfully applied for estimation of commercial tramadol products.

All the results show that the proposed method can be used as an alternative for the assay of Tramadol HCl capsules. It shows an easy sample treatment procedure consuming less time compared to other procedures such as colorimetry [7], visible and ultraviolet spectroscopy [8, 9], HPTLC [10] and HPLC [11-13]. The advantage is that no organic solvent is required. As a result, the method can be considered cost-effective and in line with green pharmacy practices.

CONCLUSION

The proposed diffuse reflectance infrared fourier transform spectroscopic method was applied for the quantification of solid-state pharmaceuticals. In the present investigation, we report the development and validation of the eco-friendly DRIFTS method for the quantification of solid-state Tramadol HCl and its successful application to commercial formulation. The proposed method was found to be precise, accurate and suitable for analysis of Tramadol HCl in capsule dosage form. Thus, the developed method has the advantage of being solvent-free, eco-friendly, cost-effective and involving relatively simple sample preparation techniques.

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Nil

AUTHORS CONTRIBUTIONS

We declare that it's an original research work carried out by V. S. G. Pravallika B. The method validations were performed by Pranati M. Dr. Usha Rani N proofread the manuscript, suggested the necessary corrections and helped in writing the manuscript.

CONFLICT OF INTERESTS

Declared none

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