



UV SPECTROPHOTOMETRIC METHOD FOR DETERMINATION OF CLEBOPRIDE IN PURE AND IN PHARMACEUTICAL FORMULATION

B. Thangabalan^{1*}, A. Elphine Prabahar¹, R. Kalaichelvi²
and P.Vijayaraj kumar³

¹Department of Pharmacy, Donbosco PG College, Pulladigunta, Guntur-522 017, India

²K.C.Reddy Institute of Pharmaceutical Sciences, Medikonduru Mandal, Guntur-522 348, India

³Bharat Institute of Pharmacy, Mangalpally Village, Ibrahimpatnam. RR Dist.-501506.

*E-mail: bthangabalan@gmail.com

ABSTRACT

A simple, sensitive and reproducible spectrophotometric method was developed for the determination of clebopride in pure form and in pharmaceutical formulation. It has an absorption maximum at 263 nm and obeys Beer's law in the concentration range 20 – 100 $\mu\text{g mL}^{-1}$. Results of analysis were validated statistically and by recovery studies. The apparent molar absorptivity and Sandell's sensitivity were $3.944 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$ and $1.06 \times 10^{-5} \mu\text{g cm}^{-2}$, respectively. The slope and intercept of the equation of the regression line are 0.0106 and 0.0045 respectively. Correlation coefficient was found to be 0.9999. This method is successfully employed for the determination of clebopride in pharmaceutical preparation.

Keywords: UV Spectrophotometry, clebopride, Tablet analysis.

INTRODUCTION

Clebopride, 4-amino-N-(1-benzylpiperidin-4-yl)-5-chloro-2-methoxybenzamide (Figure 1), is a dopamine antagonist drug with antiemetic and prokinetic properties used to treat functional gastrointestinal disorders. Detailed investigation at several centres has demonstrated its encouraging antiemetic, gastrokinetic and anxiolytic properties¹⁻³. Literature survey reveals that the drug can be estimated by thin-layer chromatography (TLC) and high-performance liquid chromatography (HPLC)⁴⁻⁶, gas chromatography-mass spectrometry (GC-MS) and radioimmunoassay (RIA) in both animals⁷ and man^{8,9}. No spectrophotometric method has been reported in the literature for the assay of clebopride. This communication describes a simple and sensitive spectrophotometric method for the determination of clebopride in bulk and drug formulations.

EXPERIMENTAL

Apparatus:

The spectrophotometric measurements were carried out using An Elico UV/Visible double beam spectrophotometer SL-164 with 1 cm matched quartz cells.

Reagents:

Clebopride was tested for purity by measuring its melting point and IR spectra and no impurities were found. Methanol of analytical grade and double distilled water were used.

Standard solution of the drug:

Accurately weighed 100 mg of clebopride was dissolved in sufficient quantity of methanol and the solution was diluted to 100 mL with distilled water to obtain 1000 $\mu\text{g/mL}$ of stock solution. This stock solution was further diluted with distilled water to obtain the working standard of 100 $\mu\text{g/mL}$. It was further diluted to obtain 20, 40, 60, 80 and 100 $\mu\text{g mL}^{-1}$ with distilled water. The absorbance was

measured at 263 nm against distilled water as blank. The calibration curve was plotted in the concentration range of 20 to 100 $\mu\text{g mL}^{-1}$ of clebopride in distilled water.

Preparation of sample solution:

For analysis of tablet formulation, forty tablets of clebopride were weighed accurately and finely powdered. An accurately weighed portion of powdered sample, equivalent to 10 mg of clebopride was dissolved in sufficient quantity of methanol, sonicated for 20 minutes; the resultant was filtered through whatman filter paper No. 41 and washed with methanol. The filtrate and washings were combined and the final volume was made to 100 mL with distilled water. The solution was suitably diluted and analyzed as given under the assay procedure for bulk samples. The results are represented in Table 2. None of the excipients usually employed in the formulation of tablets interfered in the analysis of clebopride, by the proposed methods.

RESULTS AND DISCUSSION

Analytical data:

The UV spectrum of standard solutions of clebopride in distilled water was illustrated in Figure 2. The optical characteristics such as Beer's law limits, Sandell's sensitivity, molar absorptivity and the results are summarized in Table 1. The assay and precision studies results for tablets containing clebopride are shown in Table 2.

Validation:

The assay of clebopride was validated with respect to linearity, precision and accuracy.

Linearity and range:

In developed UV method, calibration curve was linear in the range from 20 to 100 $\mu\text{g mL}^{-1}$ of clebopride.

Precision:

Inter-day precision: This was done by analyzing formulation by same analyst for six days subsequently. The % RSD values are shown in Table 2.

Intra-day precision: This was done by analyzing formulation in same day for six times of individual preparation and observation. The % RSD and datas are shown in Table 2.

Accuracy:

Recovery studies were performed to judge the accuracy of the method. Recovery studies were carried out by adding a known quantity of pure drug to a pre-analyzed formulations and the proposed method was followed. From the amount of drug found, percentage recovery was calculated. The results of analysis and recovery studies are given in Table 3.

CONCLUSION

The developed UV method is found to be simple, sensitive, accurate, precise and most reproducible and can be used for the routine quality control analysis of clebopride in bulk drug and its formulations.

Table -1:Optical characteristics of proposed method

Parameters	Values
λ_{max} (nm)	263
Beer's law limit ($\mu\text{g mL}^{-1}$)	20-100
Sandell's sensitivity ($\mu\text{g cm}^{-2}/0.001$ absorbance unit)	1.06×10^{-5}
Molar absorptivity ($\text{L mol}^{-1} \text{cm}^{-1}$)	3.944×10^3
Regression equation ($Y = a + bc$)	
Slope (b)	0.0106
Intercept(a)	0.0045
Correlation coefficient (r^2)	0.9999

Table -2: Assay results and precision studies

Formulations	Labeled amount (mg/ tablet)	(%) label claim* \pm S.D	Precision**		
			Repeatability	Inter-day	Intra-day
Clebopride Tablets	0.5	99.96 \pm 0.0104	0.4591	0.6587	0.4667

* Average of six determinations. **SD of five determinations.

Table -3: Recovery study

Drug	Label Claim (mg/ tablet)	Spike level (%)	Amount of drug added (mg)	Amount of drug recovered (mg)	Percentage recovery \pm SD*
Clebopride Tablets	0.5	80	8.0	7.99	99.98 \pm 0.6834
		100	10.0	10.02	100.16 \pm 0.7432
		120	12.0	12.06	100.18 \pm 0.3788

*Mean of six determinations.

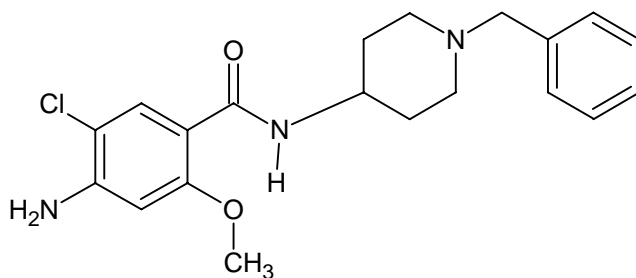


Fig.- 1: Chemical structure of Clebopride.

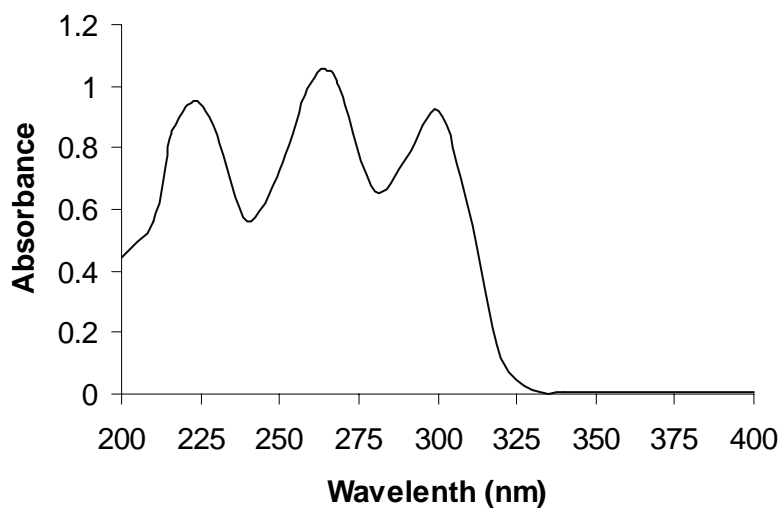


Fig.- 2: UV spectrum of Clebopride in distilled water.

REFERENCES

1. J. Prieto, J. Moragues, R. G. Spickett, A. Vega, M. Colombo, W. Salazar and D. J. Roberts, *The journal of pharmacy and pharmacology*, **29**, 147 (1977).
 2. P. N. C. Elliot, P. Jenner, G. Huizing, C. D. Marsden and R. Miller, *Neuropharmacology*, **16**, 333 (1977).
 3. D. J. Roberts, *Current therapeutic research*, (Suppl. IS), 31 (1982)
 4. G. Huizing, A. H. Beckett and J. Segura, *Journal of Chromatography*, **172**, 227 (1979)
 5. J. Segura, I. Garcia, L. Borja, E. Tarrus and O. M. Bakke, *Journal of Pharmacy and Pharmacology*, **33**, 214 (1981).
 6. D. J. Roberts, Laboratorios Almirall, Barcelona 1985, personal communication.
 7. Y. Hayasaka, S. Murata and K. Umemura, *Chemical & pharmaceutical bulletin*, **29**, 1478 (1981).
 8. M. Yano, K. Nakamichi, T. Yamaki, T. Fukami, K. Ishikawa and I. Matsumoto, *Chemical & pharmaceutical bulletin*, **32**, 1491 (1984).
- P. R. Robinson, M. D. Jones and J. Maddock, *Journal of Chromatography*, **432**, 153 (1988).

(Received: 8 August 2009

Accepted: 26 August 2009

RJC-429)

RJC will widely cover all branches of **CHEMISTRY** including: Organic, Inorganic, Physical, Analytical, Biological, Pharmaceutical, Industrial, Environmental, Agricultural & Soil, Petroleum, Polymers, Nanotechnology, Green Chemistry, Forensic, Phytochemistry, Synthetic Drugs, Computational, as well as Chemical Physics and Chemical Engineering.

Manuscript Categories: Full-length paper, Review Articles, Short/Rapid Communications.

Manuscripts should be addressed to:

Prof. (Dr.) Sanjay K. Sharma

Editor-in-Chief

23, 'Anukampa', Janakpuri, Opp. Heerapura Power Station,
Ajmer Road, Jaipur-302024 (India)

E-mail: rasayanjournal@gmail.com, drsanjay1973@gmail.com

Mobile: 09414202678, 09887050628

If you think that you may be a potential reviewer in field of your interest, write us at rasayanjournal@gmail.com with your detailed resume and recent color photograph.

Adopt **GREEN CHEMISTRY**
Save Our Planet.

We publish papers of Green Chemistry on priority.