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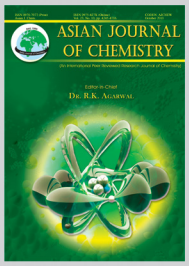

*NOTE*

# Visible Spectrophotometric Method for the Estimation of Clebopride in Pure and in Pharmaceutical Formulations

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A simple, sensitive, accurate and economic method has been developed for the quantitative estimation of clebopride in pure and in tablets. This method is based on the reaction of the drug in methanolic solution with *p*-dimethylamino benzaldehyde in acidic condition producing Schiff's base having a $\lambda_{max}$ at 444 nm. Beer's law is obeyed in concentrations ranging from 50-175 µg/mL. The results of analysis have been validated statistically and by recovery studies.

**Key Words: Visible spectrophotometry, Clebopride, *p*-Dimethylamino benzaldehyde, Schiff's base.**

Clebopride, N-(1'-benzyl-4'-piperidyl)-2-methoxy-4-amino-5-chlorobenzamide, 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 properties[1-3]. Literature survey reveals that the drug can be estimated by thin-layer chromatography and high-performance liquid chromatography[4-6], UV spectrophotometry[7], gas chromatography-mass spectrometry and radioimmunoassay in both animals[8] and man[9,10]. In the present work, clebopride in tablet formulations were analyzed spectroscopically by forming Schiff's base with *p*-dimethylamino benzaldehyde and estimated at 444 nm.

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

All chemicals were of analytical reagent grade of E. Merck. The distilled water was used to prepare all solutions. Freshly prepared solutions were always employed. Tablets containing active material were kindly supplied from local stores. 1 % *p*-dimethylamino benzaldehyde was prepared by dissolving in 2N of $H_2SO_4$.

**Preparation of standard solution:** A standard stock solution of 1000 µg/mL was prepared by dissolving 100 mg of drug in 100 mL of methanol. From this 50 mL was taken in to a 100 mL volumetric flask and volume is made upto the mark with methanol which gives 500 µg/mL.

**Assay of pure drug:** From 500 µg/mL clebopride solution aliquots of 1.0, 1.5, 2.0, 2.5, 3.0 and 3.5 mL were transferred in a series of 10 mL volumetric flask. To each of the flask 2 mL of *p*-dimethylamino benzaldehyde solution was added and kept aside for 20 min for colour development. The volume was made up to mark with methanol. The absorbance of the yellow coloured chromogen was measured at 444 nm against reagent blank (Fig. 1).

**Preparation of sample drug solution:** Twenty tablets of clebopride were weighed accurately and finely powdered. An accurately weighed portion of powdered sample, equivalent to 5 mg of clebopride was dissolved in sufficient quantity of methanol, sonicated for 20 min. 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 25 mL with methanol.

**Formulation analysis:** The above mentioned filtrate (5 mL) was taken in a 10 mL volumetric flask. To the flask 2 mL of *p*-dimethylamino benzaldehyde solution was added and kept aside for 20 min for colour development. The volume was made up to mark with methanol. The absorbance of the yellow coloured chromogen was measured at 444 nm against reagent blank. The amount of clebopride present in the sample was computed from calibration curve (Fig. 1).

The optical characteristics such as absorption maxima, Beer's law limits, molar absorptivity, the limit of detection (LOD) and limit of quantification (LOQ) and Sandell's sensitivity

TABLE-2
ASSAY RESULTS, RECOVERY AND PRECISION STUDIES

| Sample | Labeled amount (mg/tablet) | Label (%) claim* ± SD | Recovery (%) | Precision SD | |
|---|---|---|---|---|---|
| | | | | Inter-day (n = 18) | Intra-day (n = 6) |
| Clebopride | 0.5 | 99.96 ± 0.740 | 99.96-100.83 | 0.0056 | 0.0034 |

*Average of six determinations.

are presented in Table-1. The regression analysis using method of least squares was made for the slope (b), intercept (a) and correlation ($r^2$) obtained from different concentrations and results are summarized in Table-1. The results obtained for formulation analysis are given in Table-2. The precision of this method were tested by analyzing six replicates of the drug. The recovery test was performed using the standard addition method and were determined from the calibration curve (Table-2). The accuracy of the method is indicated by the excellent recovery (99.96-100.83 %).

TABLE-1
OPTICAL CHARACTERISTICS OF PROPOSED METHOD

| Parameters | Value |
|---|---|
| $\lambda_{max}$ (nm) | 444 |
| Beer's law limit (µg/mL) | 50-175 |
| Sandell's sensitivity (µg/cm$^2$/0.001 absorbance unit) | $3.8099 \times 10^{-6}$ |
| Molar absorptivity (L mol$^{-1}$ cm$^{-1}$) | $1.933 \times 10^3$ |
| Limit of detection (µg/mL) | 0.8664 |
| Limit of quantification (µg/mL) | 2.5993 |
| Regression equation (Y = a + bc) | 0.0040 |
| Slope (b) intercept (a) | -0.0122 |
| Correlation coefficient ($r^2$) | 0.998 |

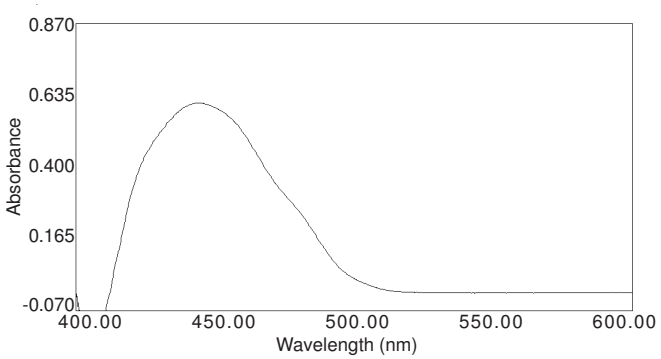


Fig. 1. Absorption spectrum of clebopride with PDAB shows $\lambda_{max}$ at 444 nm

## Conclusion

The proposed method can be used for determination of clebopride in tablets. The method is simple, accurate and precise and suitable for routine determination of clebopride in pure and in pharmaceutical formulations. The additives commonly used in formulation do not interfere with the quantification procedure.

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