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Simple UV Spectrophotometric Method for Estimation of Dicyclomine Hydrochloride in Bulk and Tablet Formulation

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Subject: Analytical Chemistry

Abstract
The aim of the present study was to develop a simple, accurate, precise, reproducible and economical UV spectrophotometric method for estimation of Dicyclomine hydrochloride (DIC) in bulk and tablet formulation. Single point standardization method was used for quantitative estimation of Dicyclomine hydrochloride. A solvent system of 0.1N HCl was used to dissolve the drug in bulk and in its dosage form. The UV absorbance was determined at 213nm (λmax of DIC). The solution of DIC obeyed Beer's law in the concentration range of 100-500 µg/ml. The method was validated for parameters such as linearity, accuracy, precision and limit of quantification, limit of detection, ruggedness and robustness as per ICH guidelines. The proposed method is thus recommended for routine analysis of DIC in bulk and tablet formulations without any interference of the excipient. Further it is a simple and rapid method for estimation of DIC which is otherwise a weakly absorbing drug in UV region.

Keywords: *Dicyclomine hydrochloride, single point standardization method, validation*

Introduction
Dicyclomine, 2- (diethyl amino) ethyl 1-cyclohexyl cyclohexane carboxylate is an antispasmodic and anticholinergic agent. It is used to treat a certain type of intestinal problem called irritable bowel syndrome. It helps to reduce the symptoms of stomach and intestinal cramping. It helps in offering relief to the cramps of stomach, bladder and intestine. ([1, 2, 3]) Figure 1 represents the structure of Dicyclomine. The drug is used either alone or in combination with other drugs for the treatment of diarrhea and dysentery of amoebic, bacterial or mixed origin. Literature survey revealed analytical methods for simultaneous estimation of DIC in combination with other drugs such as UV spectrophotometry, RP-HPLC and HPTLC. No simple zero order UV spectrophotometric method is yet reported for estimation of DIC alone and its dosage forms. However analytical methods such as spectrophotometric, ([4, 5]) RP-HPLC, ([6]) HPTLC, ([7]) spectrophotometric using Pi acceptors, NMR, ([8]) gas liquid chromatography, ([9]) potentiometry ([10]) and calorimetric ([11]) have been reported for estimation of DIC alone.

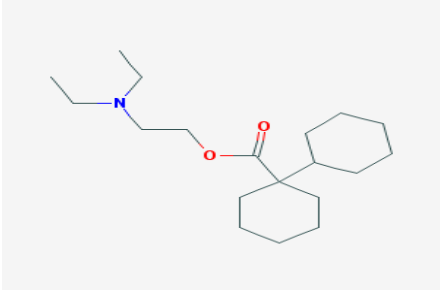

Fig 1: Structure of Dicyclomine hydrochloride [1]

Materials and Methods
Instrumentation
A double beam LABINDIA 3000$^+$ UV/Visible spectrophotometer with a pair of 1 cm matched quartz cells was used. Shimadzu digital balance (Model No: AUY220) was used for weighing the samples.

Materials and Reagents
All the chemicals were of analytical reagent grade and solutions were prepared with distilled water. Dicyclomine hydrochloride was obtained

as a gift sample from Hindustan Laboratories Pvt. Ltd. Palghar. Tablets were purchased from local pharmacy, each containing 20mg of DIC (Bentyl, Axcan Scandipharma) and were used in same ratio for preparation of calibration curves.

Analytical Method Development

Selection of solvent
Selection of solvent was based on solubility and stability of drug in solvent system as well as extraction of drug from its formulation. Dicyclomine hydrochloride is freely soluble in water and organic solvents. It was also found to be soluble in 0.1N HCl. Therefore 0.1N HCl was chosen as solvent for UV spectrophotometric determination.

Preparation of stock solution
A standard stock solution of DIC (25mg) was prepared by dissolving drug in 25 ml of 0.1N HCl to get the final concentration of 1000µg/ml of each drug.

Preparation of working standard solution
The above stock solution having concentration of 1000µg/ml was used to prepare various solutions having concentration of DIC ranging from 100-500 mcg/ml.

Determination of wavelength
The solutions were scanned in UV range from 200-400nm. The λmax value was obtained as 213 nm. The UV spectrum of DIC is shown in figure 2.

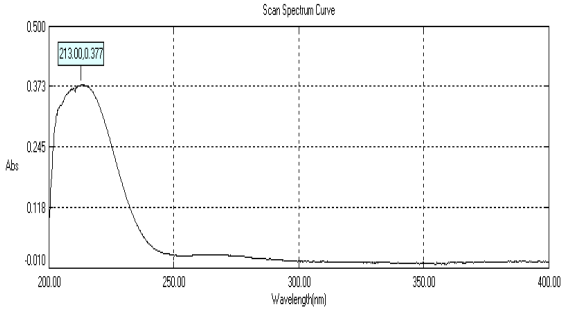

Fig 2: Zero order spectrum of DIC

Method validation

Purpose:
The method was validated for various parameters such as precision, accuracy, specificity, robustness, LOD and LOQ as per ICH guidelines. ([12])

Results and Discussion

Linearity profile
The absorbance of DIC solution having concentration ranging from 100-500 mcg/ml was measured at 213nm. The calibration curve was generated as absorbance versus concentration as shown in figure 3.

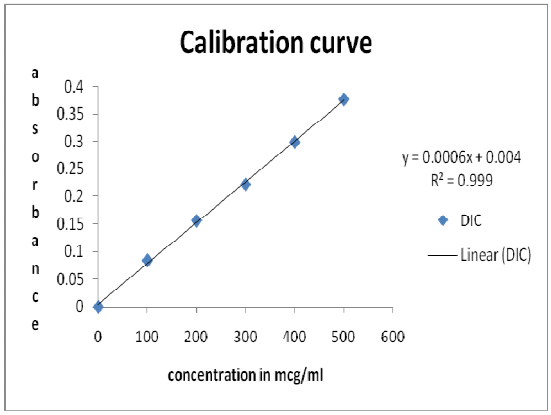

Fig 3: Calibration curve for DIC

The linearity profile of DIC is shown in table 1. The regression equation was obtained as Y = 0.0006X + 0.004 and correlation coefficient was found to be 0.999. Beer's law was obeyed in the range of 100-500µg/ml.

Table 1: Linearity profile

Parameters	Results
Slope	0.0006
Intercept	0.004
Regression equation	Y= 0.0006x+0.004
Correlation coefficient	0.999
Wavelength	213nm
Linearity	100-500 mcg/ml
LOD	9.24 mcg/ml
LOQ	28 mcg/ml

Accuracy
To check the accuracy of the developed method and to study the interference of formulation excipients, analytical recovery experiments were carried out by using three different concentrations at level of 80%, 100% and

120% such as 240 mcg/ml, 300 mcg/ml and 360 mcg/ml. 240µg/ml, 300µg/ml and 360µg/ml. From the total amount of drug found, the percentage recovery was calculated. This procedure was repeated three times for each concentration. The standard deviation and % relative standard deviation was calculated.

Table 2 shows the accuracy results. The standard deviation and % relative standard deviation was found to be within limits.

Table 2: Accuracy

Recovery level	% Recovery	SD	%RSD
80%	101.84	0.595	0.580
100%	102.42	0.475	0.460
120%	101.62	1.052	1.036

Precision

The precision of the method was confirmed by repeatability and intermediate precision. The repeatability of the method was determined by analysing a specific concentration of DIC (300mcg/ml) repeatedly (nine times). The amount of drug present in the tablet formulation was calculated. The %RSD was calculated. The intermediate precision of the method was confirmed by intraday and interday analysis i.e. the analysis of formulation was repeated for three times in the same day and on three successive days. The amount of drug was determined and %RSD was calculated. The result of precision studies such as reproducibility, intraday and interday precision is depicted in tables 3-5. The standard deviation and % relative standard deviation was found to be well within limits.

Table 3: Repeatability

Observation	Value obtained
Absorbance	0..27
Mean	0.228
SD	0.0035
%RSD	1.57

Table 4: Intraday Precision

Time	%RSD
10.00 am	1.112
1.00 pm	1.625
4.00 pm	1.150
Average %RSD	1.295

Table 5: Interday Precision

Observation	%RSD
Day 1	1.24
Day 2	1.41
Day 3	1.04
Average %RSD	1.23

Limit of Detection (LOD) and Limit of Quantitation (LOQ)

The LOD and LOQ for Dicyclomine hydrochloride was determined based on calculating the signal to noise ratio (s/n is 3.3 for LOD and 10 for LOQ). From the calibration curve the standard deviation of the Y-intercepts and slope of the regression line were used. σ is standard deviation of response (y-intercept) and S is the slope of calibration plot.

$$LOD = 3.3 \times \frac{\sigma}{S}$$

$$LOQ = 10 \times \frac{\sigma}{S}$$

Robustness

Robustness of the method was determined by carrying out the analysis under different conditions of temperature such as room temperature, wavelength conditions and variation in concentrations. The respective absorbances were noted and the result was expressed as % RSD as shown in table 6.

Ruggedness

Ruggedness of the method was determined by carrying out the analysis by different analyst and the respective absorbance of solution of DIC

having concentration as 300 µg/ml was noted. The result is expressed as shown in table 7. The developed method for estimation of DIC was found to be robust and rugged as shown in table 6 and table 7.

Table 6: Robustness

Wavelength	Concentration	Absorbance	%RSD
210	240 mcg/ml	0.180	0.96
	300 mcg/ml	0.224	1.36
	360 mcg/ml	0.268	1.14
213	240 mcg/ml	0.184	1.08
	300 mcg/ml	0.229	0.75
	360 mcg/ml	0.275	0.72
215	240 mcg/ml	0.183	1.13
	300 mcg/ml	0.228	1.26
	360 mcg/ml	0.274	0.42

Table 7: Ruggedness

Observation	Analyst 1	Analyst 2
Absorbance	0.221	0.221
Mean	0.220	0.219
SD	0.0016	0.0020
%RSD	0.727	0.913

Conclusion

The developed zero order UV spectro photometric method for the estimation of DIC was found to be simple, sensitive and economical and can be routinely used in the laboratory. This method was also validated by checking the parameters such as accuracy, precision, linearity, robustness and ruggedness. The proposed method showed high level of precision as depicted by low values of standard deviation and relative standard deviation. Hence this method can be applied successfully for the estimation of DIC in bulk and pharmaceutical formulations without interference of excipients. Although DIC is a weakly absorbing drug in UV region, it was found to have considerable absorbance when dissolved in 0.1N HCl. This method is also simple as compared to earlier methods reported for estimation of DIC alone in bulk and in dosage forms.

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