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Research Article

IR QUANTIFICATION OF CHLORTHALIDONE IN BULK AND ORAL DOSAGE FORM

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Abstract

Simple and sensitive Infrared spectrophotometric method have been developed for the estimation of chlorthalidone in tablet dosage form and the Beer's concentration range was found to be 0.5mg-2.0mg. The correlation coefficient for the method was found to be 0.9942 and the developed method was analyzed for specificity, linearity of response, precision and accuracy. Thus the proposed method could be adopted for routine analysis of bulk drug and its formulation.

Keywords: Infrared spectroscopy (IR), Potassium thiocyanate(KSCN), Potassium bromide disc.

Introduction[1-4]

Chlorthalidone is (RS)-2-chloro-5-(3-hydroxy-1-oxo isoindoli-3-yl) benzene sulphonamide and is widely used in antihypertensive pharmaceutical preparations, reduces active sodium reabsorption and peripheral vascular resistance. Chlorthalidone is a diuretic drug used to treat hypertension. It is described as a thiazide diuretic .Literature survey revealed that few sophisticated analytical methods have been reported for the estimation of chlorthalidone. The present work aims to devise a novel method using Infrared spectrophotometry (IR) which has not been reported till date.

Materials and Methods

All the chemicals used throughout the experiment were of highest purity of (IR grade).

- Potassium bromide (KBr)
- Internal standard: potassium thiocyanate (KSCN)
- Bulk material: Gift sample of chlorthalidone was obtained from Madras pharmaceuticals.
- Dosage form: Chlorthalidone tablets was purchased from local market.

Instrumentation

All spectral measurements were made on ABB-IR instrument (model no: MB 3000) with KBr press (model no:M 15)

Method

Standard preparation: Calibration of the standard: Potassium thiocyanate was used as an internal standard which was preground, dried, and then reground with dry KBr to make a concentration of about 0.2% by weight of potassium thiocyanate. The final mixture was stored over phosphorus pentoxide. Stock of standard was prepared in alcohol and diluted with ethanol to give a concentration of 100mcg/ml. From this aliquot quantity required were pipette out to meet the required concentration and evaporated in a porcelain dish. To the residue known quantity of KBr-KSCN was added mixed and homogenized using agate mortar& pestle under IR lamp (Table-1). The discs were prepared by using KBr press and the infrared spectrum was recorded in absorbance mode(Fig1).Ratio of absorbance was taken from the two wave numbers (2067.54cm^{-1} /1704.95cm^{-1}). A standard calibration curve was constructed using ratio of absorbance versus concentration and presented in Table-2, (Fig 2).

Table-1: Concentration of KBr/KSCN mixture and standard.

KBR/KSCN (in mg)	50	50	50	50	50
Standard in) (in mg)	0.0	0.5	1.0	1.5	2.0

Fig.1: IR Spectra of standard Chlorthalidone with internal standard (KBr/KSCN).

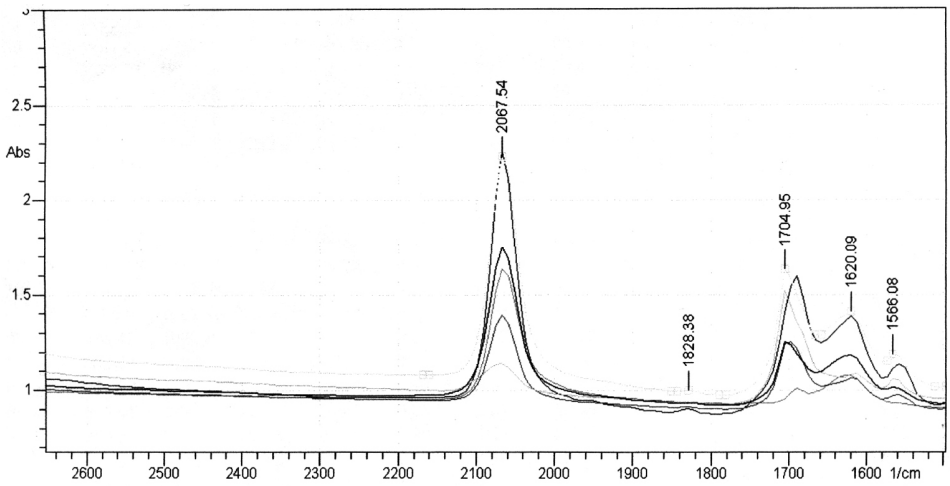
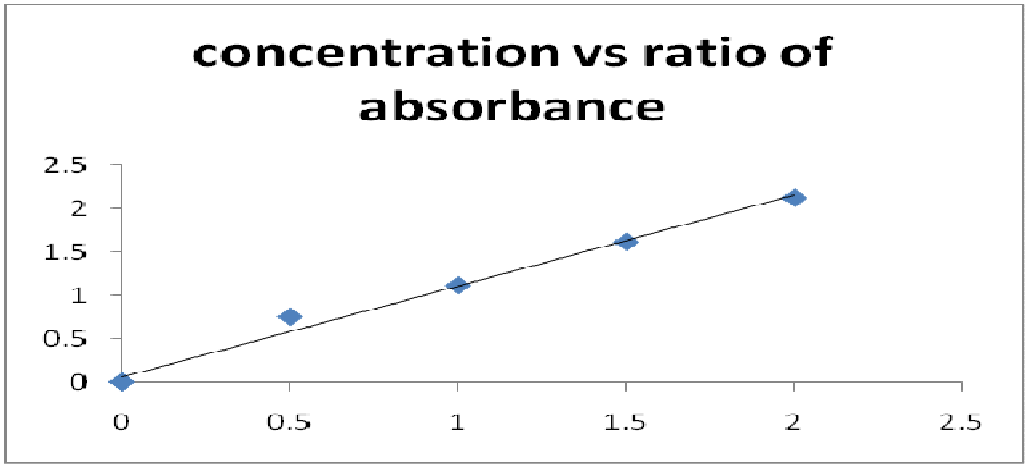


Table-2: Concentration Vs Ratio of absorbance.

concentration(in mg)	Ratio of absorbances (2067.54cm^{-1} /1704.95cm^{-1})
0.5	0.75
1	1.112
1.5	1.613
2	2.15

Fig.2 Calibration graph for chlorthalidone



Sample preparation

10 tablets of Chlorthalidone were weighed and ground to fine powder. Accurately weighed tablet powder equivalent to 10mg of chlorthalidone is dissolved in 100ml of ethanol to make a concentration of 100mcg/ml. From that 10 ml of solution which was equivalent to 1mg was taken in a porcelain dish and evaporated. Then it was mixed with the KBr/KSCN mixture and then homogenized by using agate mortar & pestle under IR lamp. The final powder was transferred to KBr press to form a disc and the infrared spectrum in absorbance mode was recorded. The sample peak area was interpolated on the respective linearity chart of the chlorthalidone and the concentration was determined.

Recovery Studies

The recovery studies were carried out on spiked samples by adding predetermined amount of standard drugs to the respective sample. About 50 and 100% of standard drugs were added to the sample and the absorbance was measured. The percentage recovery was calculated. The recovery study was performed at two levels to confirm the precision and accuracy of the above said method.

Results and Discussion

Chlorthalidone was found to obey Beer's law in the concentration range of 0.5mg-2.0mg. chlorthalidone good linearity as indicated by correlation coefficient value of to 0.9942. The optical parameters of chlorthalidone are presented in table-3. The percentage of the drug in the formulation was calculated and presented in table-4. The results of the analysis showed that the amount of drug present in the formulation was in good agreement with the label claim of the formulation. The accuracy of the proposed method was determined by recovery study. The recovery studies were carried out on spiked samples at two levels 50%, 100%. The percentage recovered were found to be in the range of 95-100% represented in table-5. The IR quantification process does not involve prior extraction and is independent of drug materials solubility.

Table-3: Optical parameters of chorthalidone by IR spectrophotometry.

Parameters	IR spectroscopy quantification method
Beer's law limit	0.5-2.0
Regression equation	1.0206x+0.0984
Slope	1.0206
Intercept	0.0984
Correlation coefficient	0.9942
Standard deviation	0.5274

Table-4: Result of tablet Assay and statistical parameters for chlorthalidone by IR spectrophotometry.

Method	**Label Claim(in mg)**	**Amount of drug found by proposed method (in mg)**	**% Label claim**	**SD**	**%RSD**
KBr disc method using Internal standard	12.5	12.25	98.0	0.5274	47.42

*The given value is a mean of 3 determinations

Table-5: Recovery study for chlorthalidone by infrared spectrophotometric method.

Method	Label claim(in mg)	Amount of drug added(in mg)	Amount of drug recovered	%recovery
IR quantification method	12.5	1.5	1.4463	96.42
		2.0	1.9064	95.32

Conclusion

The percentage recovery of the method lies between 95- 100 %. The correlation coefficient for the method was found to be 0.9942 and the recovery studies indicates that there is no interference of other ingredients present in the formulation. Thus the method is simple, precise, accurate, less time consuming and could be used for routine analysis.

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