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ANALYTICAL TECHNIQUES FOR THE ESTIMATION OF PHARMACEUTICAL DRUGS IN PURE AND TABLET DOSAGE FORMS

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ABSTRACT

A reverse phase high performance liquid chromatography (RP-HPLC) method for the analytical techniques for the estimation of pharmaceutical drug in pure and tablet dosage forms developed. Chromatography carried out 30ºC temperature on Agilent Zorbax Bonus RP (250×4.6 mm,5µ) Column. Column using Mobile Phase 0.1% Perchloric acid and ACH in ratio {60:40}) withflow rate 1ml/min. Validation parameter such as System sutability, Linearity, Recovery, Accuracy, LOD&LOQ. In that injection volume 10µl. In that HPLC wavelength is 234nm.

KEYWORDS: Chlorthalidone, RP-HPLC.

INTRODUCTION

Chlorthalidon is a prescription drug used to treat high blood pressure (hypertension). Lowering high blood pressure helps prevent strokes, heart attacks and kidney problems. It is also used to reduce extra salt and water in the body caused by condition such as heart failure, liver disease and kidney disease.

Chlorthalidone is a water pill. It increase the amount of urine you make, especially when you first start the medication. It is also helps to relax the blood vessels.

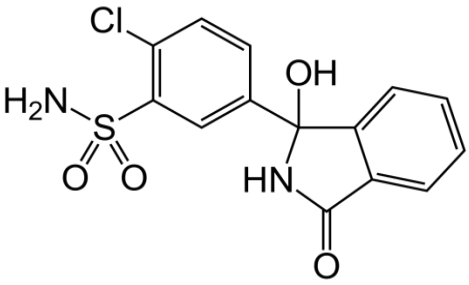


Fig no. 1: Structure of Chlorthalidone.

MATERIAL AND METHOD

Chemical And Reagent

Analytical pure sample of Chlorthadione were received as a gift sample from Aditi Pharmaceutical Private Limited were used in the study. The pharmaceutical dosage form used in this study was CTD 12.5 labeled to contain Chlorthalidione. The solvent used were of HPLC 0.1% Perchloric acid and ACH used in Preparation.

Preparation of Mobile phase

1000ml Mobile phase was prepared by mixing 600ml 0.1% Perchloric acid in water and 400ml ACH.

Apparatus and Chromatographic Condition

Chromatographic separation Agilent zorbax bonus-RP (250 × 4.6 mm, 5µ) was used for separation. The elution was carried out gradient at flow rate 1ml/min 0.1% Perchloric acid and ACH (60:40) Mobile phase.

STANDARD PREPARATION

Standard stock solution of Chlorthalidone 1 (SSS-1)

a). Initally Prepare a standardnstock solution of by adding 10 mg of Chlorthalidone in 10 ml volumetric flask & add 5 ml diluent, mix for 2 minutes and make the volume to 10 ml with diluents.(conc. Of Chlorthalidone = 1000µg/ml)

b) Then add 1.0 ml of SSS-1 in 10ml volumetric flask and add 5 ml diluent and vortex and make up volume with diluent. (Conc.of Chlorthalidone = 100 µg/ml).

Tablet Sample Preparation for Assay

a) **Tablet Sample Solution (TSS)**

1) 10 Tablets were weighed and average weights are calculated and tablets were crushed in morter and pestle.
2) Powder weight equivalent to 12.5 mg Chlorthalidone was weighed into 10 ml volumetric flask and add 5 ml diluents, Sonicate for 10 minutes and, make the volume to 10ml with diluentd. (conc.of Chlorthalidone = 1250µg/ml)

3) Pipette out 0.8 ml from above solution in a 10 ml volumetric flask and dilute upto the mark with diluents (Conc. of Chlorthalidone = 100µg/ml)

Selection of Wavelength

The sample was scanned from 200-400 nm with PDA detector. The wavelength select analysis chosen was 234 nm on basis of appropriate intensity of Chlorthalidone.

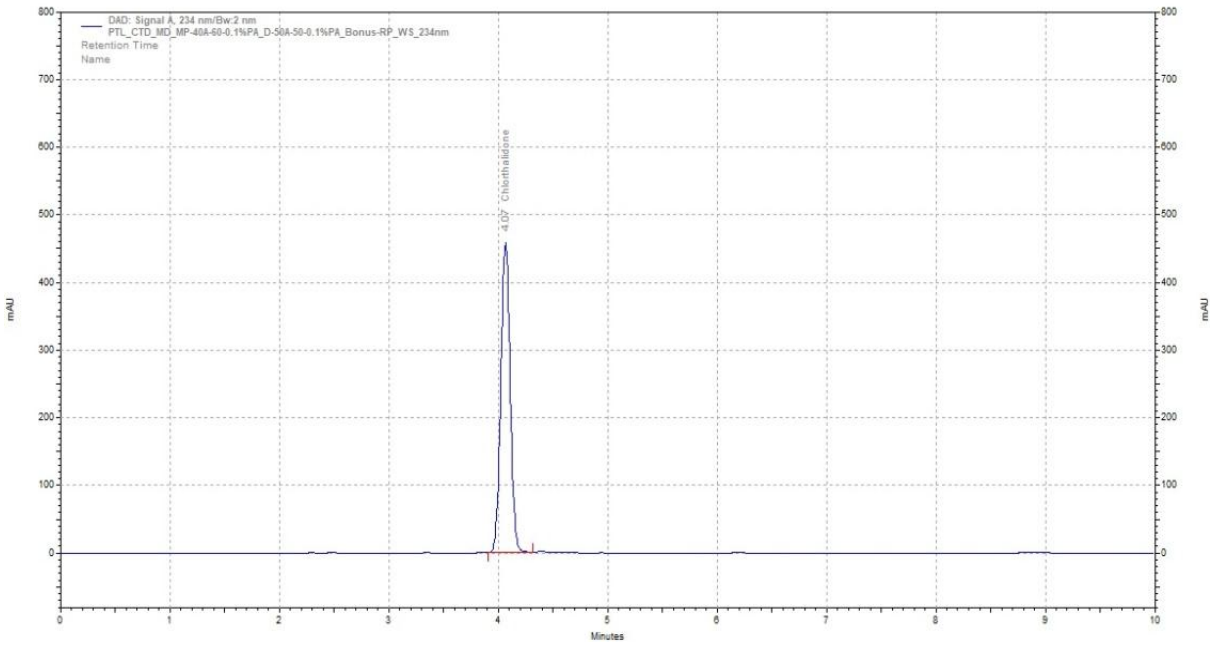


Fig. 2: Chromatogram of Standard mixture of Chlorthalidone.

Table No1: Chromatographic Condition.

Oven Temprature	30ºC
Flow Rate	1ml/min
Mobile Phase	0.1% Perchloric acid:ACH (60:40)
Run Time	08Min
Injection Volume	10µl
Wavelength	234nm
Diluent	50%-1%Perchlori Acid-50%ACH
Column	Aglent Zorbax Bonus RP(250×4.6MM,5µ)

Assay: The Assay of Chlorthalidone sample formed 101.87%.

Specificity

1) Individual samples of Chlorthalidone were prepared of 100µg/ml, Respectively and peaks were for identified from Retention time.
2) Blank was injected to ensure there is no blank peak interfering with the main analyte peaks.

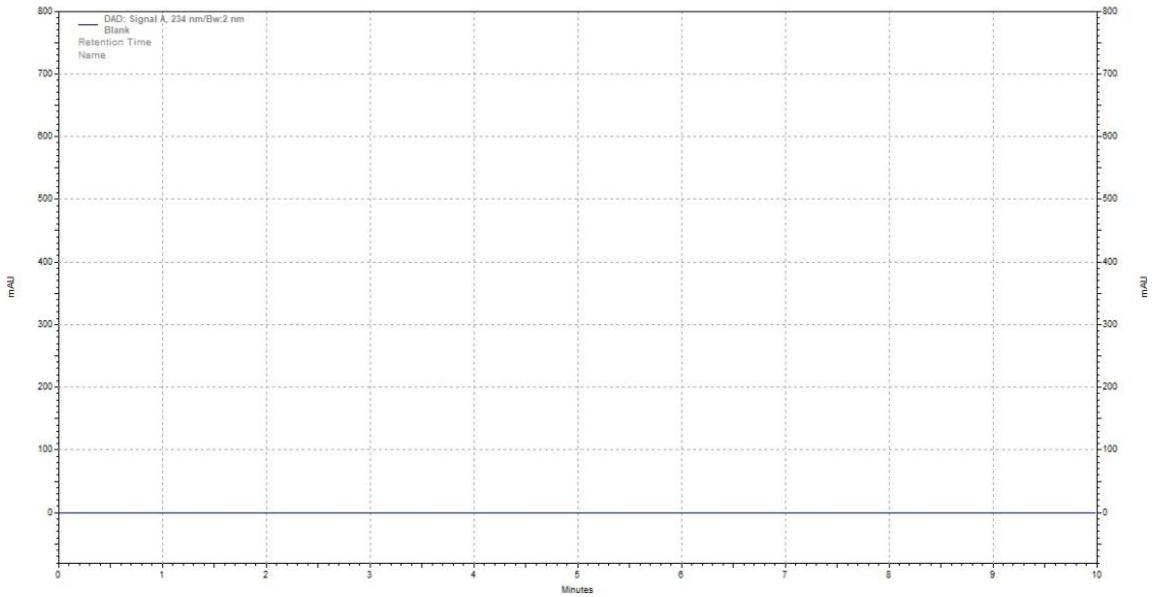


Fig. 3: Assay of Chlorthalidone.

METHOD VALIDATION

Linearity

1) 5 sample of varying concentration ranging from 80-120 were made.
2) The concentration are given below:
3) The sample preparation are given as below:
4) X ml of Chlorthalidone were added to 10ml diluents to make up the concentrations given above

Table No: 2.

X ml Of SSI	Diluted
0.8	10
0.9	10
1	10
1.1	10
1.2	10

Accuracy

1) Samples were prepared of 80%, 100% and 120% concentration by spiking the same amount of concentration given above table for Chlorthalidone.
2) Samples were injected in duplicate to calculate % RSD.
3) % Recovery was also calculated.

System Suitabiity

1) A single sample was prepared as described and 5 injections were made from same sample and checked for system suitability.

Precision

The precision of the method was done by system precision. The percentage RSD value was found to be within the limit. The percentage RSD value for peak area ratio of Chlorthalidone obtained, thus the result showing that equipment used for the work. In that precision method mainly 5 samples are formd.

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

1) It Was calculated for both drugs by using ANOVA technique
2) Formula:

$$LOD = \frac{3.3 \times \text{Std. Error of Intercept}}{\text{Coefficients of X Variable 1}}$$

$$LOQ = \frac{10 \times \text{std. Error of Intercept}}{\text{Coefficients of X variable 1}}$$

RESULT AND DISCUSSION

Assay of Chlorthalidone

Assay was found to be 101.87% of Chlorthalidone in below table:

Table No: 3.

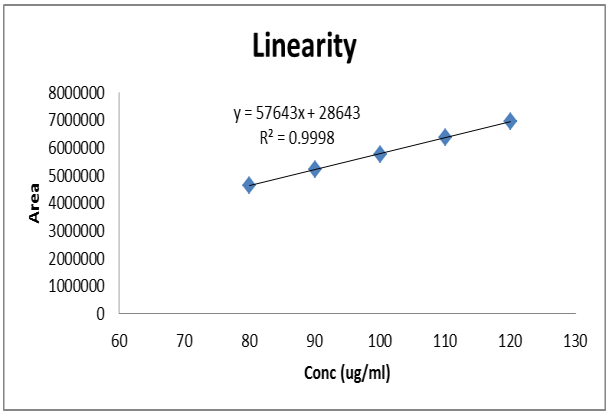
Sample	Area	Assay
WS	5770560	-
DP	5878723	101.87

Linearity of Chlorthalidone

5 Sample of varying concentration ranging from 80-120were made. The Concentration are given below:

Table No: 4.

% Level	Chlorthalidone Conc(µg/ml)	Area
80	80	4642278
90	90	5224638
100	100	5773415
110	110	6375593
120	120	6948968



Precision

The precisionof the Chlorthalidone method was found to be good with % RSD less than 2, Indicate the method was precise and the results presented below table.

Table No: 5.

Sample ID	Area
Rep 1	57733415
Rep 2	5780404
Rep 3	5769229
Rep 4	5769646
Rep 5	5760106
Average	5770560
STDEV	7364.5847
RSD	0.13

Accuracy

In accuracy study percentage recovery range of Chlorthalidone 100.56% to 100.100.50%. The range of % RSD is 0.07 % to 0.11%.

Table No: 6.

Sample ID	Reps	Spiked Conc(µg/ml)	Area	Amt Recovered(µg/ml)	% Recovery	Average	STDEV	RSD
80%	Rep 1	79.976	4642278	80.42	100.56	100.51	0.072909	0.07
	Rep 2	79.976	4637518	80.34	100.46			
100%	Rep 1	99.97	5773415	100.02	100.05	100.11	0.085641	0.09
	Rep 2	99.97	5780404	100.14	100.17			
120%	Rep 1	119.964	6948968	120.38	100.35	100.43	0.108057	0.11
	Rep 2	119.964	6959550	120.57	100.50			

System Suitability

Table No 7.

Sample ID	RT	TP	Asymmetry
Rep 1	4.07	10350	1.02
Rep 2	4.07	10292	0.99
Rep 3	4.07	10370	0.99
Rep 4	4.06	10279	1.05
Rep 5	4.07	10407	1.02
Average	4.068		
STDEV	0.004472		
RSD	0.11		

LOD & LOQ

LOD & LOQ of Chlorthalidone is 2.36µg/ml & 7.16 µg/ml.

Table No: 8.

LOD	2.36	µg/ml
LOQ	7.16	µg/ml

CONCLUSION

It includes that the developed method is simple, accurate and precise and suitable for the routine analysis, The developed methods were validated as per ICH guidelines and were found to be within limit.

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