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UV spectrophotometric determination of chlorthalidone in tablet dosage form by using single point standardization method

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Abstract

The current research endeavors to elucidate the creation of an uncomplicated, highly sensitive, swift, precise, and cost-effective UV-accepted spectrophotometric method for the quantitative assessment of Chlorthalidone. This is achieved through the utilization of a visible spectrophotometric approach employing single-point standardization and calibration plot methods, for pharmaceutical dosage forms. The equipment employed includes a double-beam UV-visible spectrophotometer, specifically the Shimadzu Model UV1800, with 1cm quartz cells and 0.2 M Sodium hydroxide serving as the solvent. Notably, an absorption maximum is identified at 219 nm. The developed method strictly adheres to Beer's law.

In the case of single-point standardization, the percentage of Chlorthalidone detected falls below the labeled claimed limit. Simultaneously, the tablet formulation is subjected to a percentage purity test using the calibration plot method, revealing that the observed quantity of Chlorthalidone is below the labeled content. This suggests a potential discrepancy in the marketed product of Chlorthalidone, indicating a probable deficiency in the therapeutic effect of the formulation due to the lower amount of Chlorthalidone present. The overall efficacy of the product hinges on the quality assurance of its constituents.

Keywords: Chlorthalidone; Single point standardization; Calibration plot method; UV; Visible spectrophotometer

1. Introduction

UV absorption spectroscopy deals with absorption of light by sample in the Ultra Violet (UV) region between wavelengths 190-380 nm while UV-Visible absorption spectrophotometry (colorimetry) deals with absorption of light by sample in the visible region between 380-780 nm. Absorption of UV-Visible light causes promotion of a valence electron from bonding to antibonding orbitals. The wavelength at which the maximum absorption bands occur will give information about the structure of the molecule or ion and the extent of the absorption is proportional with the amount of the species absorbing the light. It is used for both qualitative and quantitative investigation.

Chlorthalidone increases the excretion of sodium, chloride, and water into the renal lumen by inhibiting sodium ion transport across the renal tubular epithelium. Its primary site of action is in the cortical diluting segment of the ascending limb of the loop of Henle. Thiazides and related compounds also decrease the glomerular filtration rate, which further reduces the drug's efficacy in patients with renal impairment (e.g. renal insufficiency). By increasing the delivery

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of sodium to the distal renal tubule, chlortalidone indirectly increases potassium excretion via the sodium potassium exchange mechanism (i.e. apical ROMK/Na channels coupled with baso lateral NKATPases). This can result in hypokalemia and hypochloremia as well as a mild metabolic alkalosis; however, the diuretic efficacy of chlortalidone is not affected by the acid-base balance of the patient being treated.

Literature survey reveals that there are various analytical methods for estimation of chlorthalidone individually or in combination with other drugs, that involves spectrophotometric, fluorimetric methods, but not by single point standardization method.

2. Materials and methods

2.1. Instrument

UV Spectrophotometer (SHIMADZU UV 1800) and a pair of quartz cuvettes having 1cm path length was used, Digital balance, sonicator.

2.2. Chemicals

Standard Chlorthalidone was obtained as gift sample from Macleod's pharmaceutical company, Mumbai. Chlorthalidone 12.5 mg Tablet, NAOH, Distilled Water. used throughout experimental work.

2.3. Methods

Single point standardization: The single point involves the measurement of the absorbance of the sample solution of the reference substance.

$$CT = AT / AS\ CS$$

Where CT = Concentration of test solution AT = Absorbance of test solution AS = Absorbance of standard solution CS = Concentration of standard solution.

2.4. Experimental work

2.4.1. Preparation of standard solutions

Weigh 0.1 gm powdered drug of Chlortholidone and add 100 ml of 0.1 M Sodium Hydroxide solutions (1000 µg/mL). This standard solution pipette out the 5 ml solution in 50 ml cleaned volumetric flask and volume make up to the mark with the help of 0.2 M Sodium hydroxide solutions (100 µg/mL). Prepare stock solution from the standard solution pipette out the 1 ml standard solution in a 10 mL previously cleaned volumetric flask and volume make up to the mark with the help of 0.2 M Sodium hydroxide solutions (10µg/mL).

Preparation Standard Calibration Curve From prepared stock make working solution in a series of 2 ,4,6,8, 10µg/mL using prefiltered solution of 0.2 M Sodium hydroxide solutions. Take absorbance of different working solution at 219 nm. 3. Plot the graph between for obtained absorbance (nm) and concentration of different

2.5. Methods of Estimation

2.5.1. Method A (Single point standardization)

Weigh a 15 tablet of Chlortholidone crush it in powder form in a previously cleaned mortal and pastle. Powdered Chlortholidone weight 0.06 g of equivalent to Chlortholidone. Add 60 ml of 0.2 M Sodium hydroxide. This standard solution pipette out the 5 ml solution in 50 ml cleaned volumetric flask and volume make up to the mark with the help of 0.2 M Sodium hydroxide solutions(100 µg/mL). Prepare stock solution from the standard solution pipette out the 1 ml standard solution in a 10 mL previously cleaned volumetric flask and volume make up to the mark with the help of 0.2 M Sodium hydroxide solutions (10µg/mL). Resulting solution to 10 mL of 0.2 N Sodium hydroxide solution scan in ultraviolet range UV Spectrophotometer in the 200 to 400 nm.

$$CT = AT / AS\ Cs$$

Where…. CT = Concentration of test solution
AT = Absorbance of test solution

AS = Absorbance of standard solution
CS = Concentration of standard solution

$$CS = 0.0075\ \mu g/ml.$$

2.5.2. Method B (Calibration Plot Method)

From prepared stock make working solution in a series of 2 ,4,6,8, 10µg/mL using prefiltered solution of 0.2 M Sodium hydroxide solutions. Take a absorbance of different working solution at 220 nm. Plot the graph between for obtained absorbance (nm) and concentration of different and plot the graph concentration VS. Absorbance.

3. Results and discussion

Conducting an assessment of Chlorthalidone in tablet dosage forms, a UV spectrophotometric method incorporating UV-visible Chlorthalidone was executed. Standard and sample solutions were meticulously prepared, and their respective absorbances were documented. Subsequently, the absorbance maxima were computed. The analytical outcomes demonstrated a commendable concordance between the measured drug quantities and the label claim stipulated for the formulation. Notably, the tablet exhibited percentage purity values within the range of 79.46%.

In this current investigation, the quantification of Chlorthalidone in solid dosage forms was explored. Tablets of Chlorthalidone were procured from the local market, encompassing various manufacturers. Employing a UV spectrophotometer, two distinct methods were applied to scrutinize these tablet samples. The UV scan of the standard Chlorthalidone solution, conducted within the wavelength range of 200–400nm, revealed an absorption maximum at 219nm. Verification of Beer's law ensued from a calibration curve plotting observed values within the concentration range of 2-10 µg/ml, displaying a clearly linear relationship (y = 0.024x + 0.019) with a correlation coefficient of 0.969.

Through single-point standardization, Chlorthalidone content was estimated in sample A, revealing the highest amount at 79.46%. Meanwhile, employing the Calibration plot method for percentage purity assessment showcased results within the 100% range. Notably, the Calibration plot method demonstrated superior accuracy in comparison to the single-point standardization method.

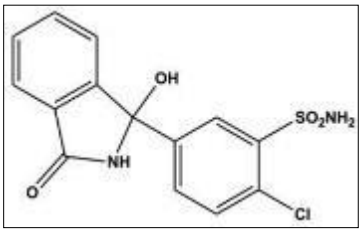

Figure 1 Chemical structure Chlorthalidone

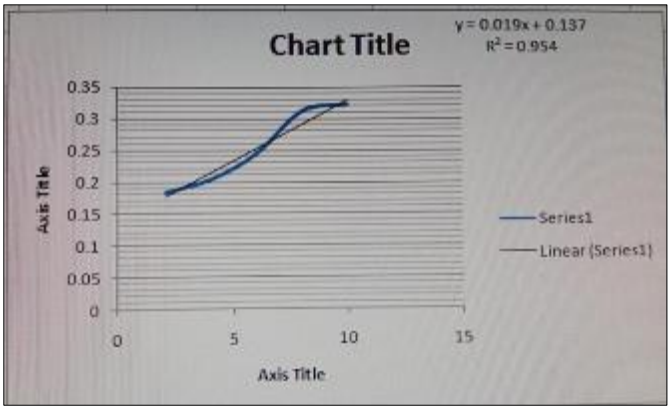

Figure 2 Standard Calibration curve of Pure Chlorthalidone

Figure 3 Absorbance maxima of Chlorthalidone at 219nm

Table 1 Absorbance of Chlorthalidone at 219nm

Sr.No	Concentration (ppm)	Absorbance
1	2	0.068
2	4	0.132
3	6	0.144
4	8	0.215
5	10	0.270

Table 2 Absorbance of Chlorthalidone at 219nm (by single point standardization method)

Sr. No	Concentration (ppm)	Absorbance
1	2	0.183
2	4	0.204
3	6	0.245
4	8	0.312
5	10	0.322

Table 3 Results by single point Standardization method

Sample code	AT	As	CT	Assay
A	0.832	1.047	7.94	79.46%

Table 4 Result by Calibration plot method

Sample code	Unknown Concentration (y)	Slope(m)	Concentration (x)	Percentage purity
A	0.158	0.02	7.94	100%

4. Conclusion

Based on the findings of this study, it can be concluded that the Chlorthalidone content in tablets aligns with the labeled claim. In the methodology employed, the calibration plot method exhibited superior accuracy when compared to the single-point standardization approach. This suggests that the marketed Chlorthalidone product is likely safe for consumption and demonstrates a positive therapeutic effect.

In the course of this investigation focused on estimating Chlorthalidone in solid dosage forms, tablets were sourced from the local market. The UV scan of the standard Chlorthalidone solution within the wavelength range of 200–400nm disclosed an absorption maximum at 219nm. The confirmation of Beer's law ensued from the calibration curve, plotting observed values within the concentration range of 2-10 µg/ml. The resulting plot depicted a clear linear relationship (y = 0.024x + 0.019) with a correlation coefficient of 0.969. Notably, the Chlorthalidone content estimation via single-point standardization in sample A revealed the highest amount at 79.94%.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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