

Difference Spectrophotometric Method for the Estimation of Caffeine Citrate in Bulk Drug

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ABSTRACT

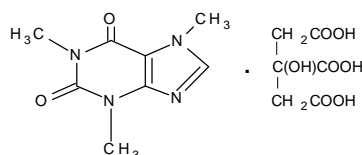
A simple, precise and sensitive UV method has been developed for the estimation of Caffeine Citrate in bulk drug form by Difference Spectrophotometric method. Caffeine Citrate has exhibited maximum absorbance at about 269 nm and 272 nm in acidic and basic solution respectively. Beer's law was obeyed in the concentration range of (2.5 -15) mcg/ml in both the cases. The proposed method was successfully applied for the determination of Caffeine Citrate in bulk drug. As per ICH guidelines the results of the analysis were validated statistically and were found to be satisfactory.

Keywords: Caffeine Citrate, Difference Spectrophotometry, Bulk, Validation.

INTRODUCTION

Caffeine citrate is chemically 1,3,7-trimethylpurine-2,6-dione;2-hydroxypropane-1,2,3-tricarboxylic acid. Caffeine citrate is a central nervous system stimulant. It also has effects on the lungs and metabolism. Caffeine citrate is used in short-term treatment of apnea of prematurity and also to treat severe migraines.¹ It is aqueous soluble & shows different wavelength in acidic & basic condition. The essential feature of difference spectrophotometric assay is that the measured value is the difference in absorbance between two equimolar solutions of the analyte in different chemical forms which exhibit different spectral characteristics & follows Beer's law.²

STRUCTURE



Caffeine Citrate

C₁₄H₁₈N₄O₉ Mol.Wt. 386.31

Objective

Caffeine citrate shows improved absorbing interference by the technique of difference spectrophotometry. Thus the objective of the present study was to develop new analytical difference spectrophotometry method & its validation parameters for the proposed method according to ICH guidelines for the estimation of Caffeine citrate bulk drug.

EXPERIMENTAL METHODS chemicals and Reagents

Caffeine Citrate [Bulk Drug] used were of Analytical Reagent grade purchased from Research Lab fine Chem. Industries Mumbai, India, Sodium hydroxide and 1N Hydrochloric acid were purchased from Poona chemical laboratory and Double distilled water was used throughout the analysis.

Instrumentation

A JASCO V-530 UV/VIS double beam spectrophotometer with 1cm matched quartz cells was used for all spectral measurements.

Selection of Common Solvent

1N HCl and 1N NaOH were selected as a common solvent for developing spectral characteristics of drug.

Preparation of Solution

Standard stock solution containing Caffeine Citrate was prepared by dissolving 100 mg in 100 ml of Distilled water and then diluted with 1N NaOH and 1N HCl separately to get series of dilution ranging from 2.5-15 mcg/ml and then absorbance recorded at 272 nm and 269 nm respectively against reagent blank. Calibration curve was prepared by plotting concentration versus difference in absorbance and found to be linear in the concentration range of 2.5-15.0µg/ml

RESULTS AND DISCUSSION

The optical characteristics such as Beer's law limits, percent relative standard deviation and % range of error were found to be within the limits and satisfactory. All of the analytical validation parameters for the proposed method were determined according to ICH guidelines. The method was found to provide high degree of precision and reproducibility. The recovery studies showed that the results were within the limit indicating no interference. The proposed method is simple, sensitive, accurate and precise and can be successfully employed for the routine analysis of the Caffeine Citrate in bulk drug.

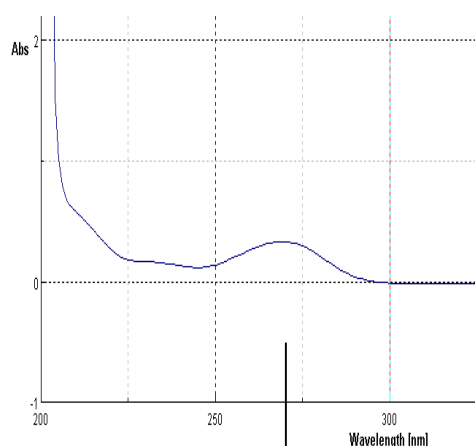


Fig. 1: 1 N NaOH with λ Max 272nm

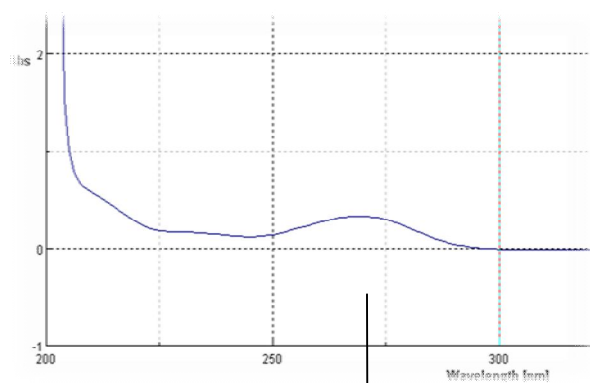


Fig. 2: 1 N HCl with λ Max 269nm

Table I: Linearity of Caffeine Citrate by Difference Spectrophotometry

S. No	Concentration Of Caffeine Citrate ($\mu\text{g/ml}$)	Absorbance at 269 nm (1N HCl)	Absorbance at 272 nm (1N NaOH)	Difference in Absorbance
1	2.5	0.0906	0.0530	0.0376
2	5	0.1785	0.1166	0.0619
3	7.5	0.2562	0.1683	0.0879
4	10	0.3348	0.2320	0.1048
5	12.5	0.4275	0.3020	0.1255
6	15	0.4981	0.3441	0.1540

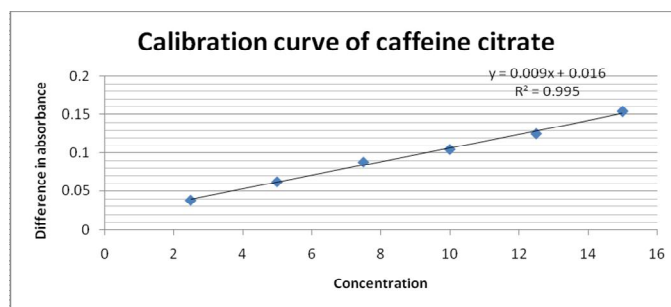


Fig. 3: Calibration curve of caffeine citrate

Table II: Characteristics and validation parameters of Caffeine citrate

Parameters	Values	
	NaOH (272nm)	HCl (269nm)
Beers's law limit ($\mu\text{g/ml}$)	2.5 -15	2.5 -15
λ_{max} (nm)	272	269
Regression equation ($Y=a + bc$)	$y = 0.009x + 0.016$	
Correlation coefficient (r)	0.995	
Slope (b)	0.009	
Intercept (a)	0.016	
Linearity	0.995	
Accuracy range (%)	98.123-100.451	
Precision	0.534	
LOD($\mu\text{g/ml}$)	0.215	
LOQ($\mu\text{g/ml}$)	0.652	

* $Y=mx+c$, where x is the concentration in ($\mu\text{g/ml}$) and Y is absorbance unit(ΔA)

CONCLUSION

The proposed method is simple, accurate, precise and selective for the estimation of caffeine citrate in bulk drug. The method is economical, rapid and do not require any sophisticated instruments contrast to chromatographic method. Hence it can be effectively applied for the routine analysis of caffeine citrate in bulk drug.

ACKNOWLEDGMENT

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