e-ISSN: 2249 – 7781
Print ISSN: 2249 – 779X

International Journal of Pharmaceutical Research & Analysis
www.ijpra.com

Research article

SIMULTANEOUS ESTIMATION OF ERGOTAMINE AND CAFFEINE IN THEIR TABLET FORMULATION

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ABSTRACT

Caffeine is most commonly used to improve mental alertness, but it has many other uses. Caffeine is used by mouth or rectally in combination with painkillers (such as aspirin and acetaminophen) and a chemical called ergotamine for treating migraine headaches. Ergotamine is in a class of medications called ergot alkaloids. It works together with caffeine by preventing blood vessels in the head from expanding and causing headaches. This combination medication is used to treat or prevent certain types of headaches (vascular headaches including migraine and cluster headaches). The developed UV-Visible Spectrophotometric method for the simultaneous estimation of ergotamine and caffeine in the tablet dosage form in the solvent system methanol and distilled water 1:1 ratio give proper estimation of percentage label claim of marketed product.

Keywords: Ergotamine, Caffeine, Estimation, Simulataenous.

INTRODUCTION

Caffeine is most commonly used to improve mental alertness, but it has many other uses. Caffeine is used by mouth or rectally in combination with painkillers (such as aspirin and acetaminophen) and a chemical called ergotamine for treating migraine headaches [1,2]. It is also used with painkillers for simple headaches and preventing and treating headaches after epidural anesthesia. Caffeine is one of the most commonly used stimulants among athletes. Taking caffeine, within limits, is allowed by the National Collegiate Athletic Association (NCAA). Urine concentrations over 15 mcg/mL are prohibited. It takes most people about 8 cups of coffee providing 100 mg/cup to reach this urine concentration. The combination of ergotamine and caffeine is used to prevent and treat migraine headaches. Ergotamine is in a class of medications called ergot alkaloids. It works together with caffeine by preventing blood vessels in the head from expanding and causing headaches. This combination medication is used to treat or prevent certain types of headaches (vascular headaches including migraine and cluster headaches). Headache pain can sometimes be caused by widened blood vessels in the head. Ergotamine works by narrowing these widened blood vessels [3,4].

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REAGENTS

All the reagents in this assay along with triple distilled water were of analytical grade. Ergotamine and caffeine were obtained from SD fine chem Ltd.

APPARATUS

Spectral analysis were made on a Jasco Spectrophotometer, Model- V-630 (Japan), was employed with spectral bandwidth of 1nm and wavelength accuracy of ±0.3nm with automatic wavelength correction with a pair of 10mm quartz cells. Glass wares used in each procedure were soaked overnight in a mixture of chromic acid and sulphuric acid rinsed thoroughly with double distilled water and dried in hot air oven.

METHODOLOGY

Validation Linearity Working standard solution of ergotamine and caffeine was taken in different 10 ml volumetric flasks and diluted up to mark with distilled water to obtained concentrations 50, 60, 70, 80, 90µg/ml of ergotamine and 2, 4, 6, 8, 10µg/ml of caffeine. A calibration curve was constructed by plotting concentration versus absorbance and line equation was calculated for both the drugs [5].

Preparation of stock solution

Accurately weighed ergotamine and caffeine (10 mg each) was transfer two separate 100ml volumetric flask, dissolved in 50 ml methanol and make up the volume up to the mark. A stoke solution contained 100µg/ml of ergotamine and caffeine [6].

Preparation of working standard

Take required quantity of 100µg/ml stock solution of ergotamine and caffeine and diluted with distilled water to obtained working standard of both solution.

Selection of detection wavelength Solutions of drug were scanned over the range of 200-400nm. It was observed that both the drug showed considerable absorbance at 278nm for ergotamine and 280nm for caffeine was selected as the wavelength for detection

Precision

The repeatability studies were carried out by estimating response of ergotamine (60µg/ml) and caffeine(4µg/ml) five times and results are reported in terms of relative standard deviation. The intermediate precision were carried out by estimating the corresponding responses 3 times on the same day and 3 different days for 3 different concentrations of ergotamine (50,60,70 µg/ml) and caffeine (4,6,8 µg/ml) and results are reportred in terms of relative std. deviation [7].

Accuracy

Recovery studies of ergotamine and caffeine were performed to judge the accuracy of the method by standard additions at three different levels 80, 100, 120 %. Mean percentage recovery was determined. Recovery values were calculated shown in table 1.

Assay of Drug Formulation

Tablets containing 5mg ergotamine Hydrochloride and 500mg caffeine Hydrochloride were taken and performed the Weight Variation Test as per I.P. These 20 tablets were weighed accurately and finely powdered. Tablet powder equivalent to 10mg ergotamine and caffeine was taken and dissolved in mixture of 50ml methanol and 50ml distilled water in 100ml volumetric flask. Sonicated this solution for 30 minutes and filter the solution. From this solution prepare working solution and the percentage content of the drugs has been found out [8, 9].

Detection Limit

The detection limit of an individual analytical procedure is the lowest amount of analytic in a sample which can be detected but not necessarily quantitative as an exact value. LOD = 3.3σ/S Where, σ = Relative std. deviation of the response, S = slope of calibration curve. Quantitation Limit The quantitation limit of an analytical procedure is the lowest amount of analyte in a sample, which can be quantitatively determine with suitable precision and accuracy. LOQ = 10σ/S Where, σ = Relative std. deviation of the response, S = slope of calibration curve.

RESULTS & DISCUSSION

The developed UV-Visible Spectrophotometric method for the simultaneous estimation of ergotamine and caffeine was found to be simple and useful with high accuracy, precision, LOD, LOQ as per ICH guidelines. Sample recoveries in all formulations using the above method was in good agreement with their respective label claim or theoretical drug content, thus suggesting the validity of the method and non interference of formulation excipients in the estimation. In the selected solvent system methanol and distilled water, drugs were stable for more than 48 hours, thus suggesting that samples need not be estimated immediately after collection. The method was successfully used for determination of drugs in their pharmaceutical formulation.

Table 1: Recovery and assay analysis

	Ergotamine	Caffeine
Amount used	50mcg	5mcg
Amount recovered	49.80mcg	4.99mcg
Percentage recovered	98.65%	99.75%
Label Claim	500mg	50mg
Estimated	498.54mg	49.57mg
Percentage	98.96%	99.67%

Table 2: LOD and LOQ analysis

S. no.	Parameters	Ergotamine	Caffeine
1	max(nm)	278	280
2	linearity range	15-39µg/ml	16-41µg/ml
3	regression equation	Y=0.0674X-0.0689	Y=0.0728X-0.0791
4	correlation coefficient	0.998	0.999
5	slope	0.0623	0.0746
6	intercept	0.0512	0.0635
7	Limit of detection(µg/ml)	0.9123	0.9872
8	Limit of quantification(µg/ml)	4.1658	5.3751
9	Intra day	0.8790±0.0417	0.9123±0.0520
10	Interday	0.8432±0.0099	0.8716±0.0154

CONCLUSION

The developed UV-Visible Spectrophotometric method for the simultaneous estimation of ergotamine and caffeine in the tablet dosage form in the solvent system methanol and distilled water 1:1 ratio give proper estimation of percentage label claim of marketed product.

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