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RP - HPLC METHOD FOR ESTIMATION OF PARACETAMOL FROM PHARMACEUTICAL FORMULATION FEBRINIL

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ABSTRACT

A simple, rapid and sensitize high performance liquid chromatography method for determination of paracetamol in a commercial dosage from Febrinil has been developed. The SHIMADZU LC 20 AT dual pump chromatograph system used a reverse phase C-18, column phenomenex C 18 5 µ, 250 mm x 4.6 mm, utilizing a mobile phase of acetonitrile / water (60 : 40 v/v/) at a flow rate of 1 mL/ min with UV detection at 210 nm. The retention time for paracetamol was found to be 2.690 min. and recovery from the formulation was 90%. The developed method was validated in terms of accuracy, precision, linearity, limit of detections, limit of quantification. This method can be used for estimation of paracetamol in pharmaceutical formulation.

Key words: RP – HPLC method, Paracetamol, Febrinil.

INTRODUCTION

Paracetamol (acetaminophen) is chemically 4- hydroxyl acetanilide. It is a centrally and peripherally acting non-opioid analgesic and antipyretic drug. Paracetamol is available in market in different dosage forms such as tablets, capsules, drops, elixirs, suspension and suppositories Literature survey revealed few HPLC and spectrophotometric methods[1-3] Paracetamol is a major metabolite of phenacetin and has similar analgesic, antipyretic action. It is a weak inhibitor of prostaglandin synthetase and is as potent as aspirin in inhibiting the brain prostaglandin synthetase It is a commonly used analgesic and antipyretic drug present in different pharmaceutical formulations. Various combination products, of Paracetamol are available and marketed. Tramadols hydrochloride (±) trans-2-(dimethylamino) methyl-1-(3-methoxyphenyl) cyclohexanol is a synthetic centrally acting analgesic agent, used for the relief. Of moderate to chronic pain and has no clinically relevant cardiovascular or respiratory depressant activity[4]. This combination has demonstrated genuine surgery in animal studies, with combination with paracetamol gives rapid onset of action while tramadols prolonged analgesic effect. Numerous studies have confirmed the efficacy and tolerability of Paracetamol plus tramadols in both acute and chronic pains. A few methods have been developed for the quantitisation of individual drug tramadaol by HPLC[5-7]. A tablet formulation Etoricoxib has been used in

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clinical practice analyzed by HPLC[8-10] and spectrophotometric methods. This work attempted for the estimation of paracetamol in the dosage form and methods was validated following ICH guidelines[11-12].

EXPERIMENTAL

The HPLC analysis was performed on SHIMADZU LC 20 AT dual pump system with UV at ambient temperature. A Phenomenex C_{18} Column (250 mm x 4.6 mm 5 µ) was utilized. All the calculations concerning the quantitative analysis were performed with external standardization by the measurement of peak area that are integrated automatically by computer using spinchrom CFR software program .

Material and chemicals

Pharmaceutical grade paracetamol was procured from Emcure pharmaceutical Ltd. Pune HPLC grade acetonitrile procure from Fisher scientific pune and HPLC grade water from LOBA Chemicals pharmaceutical formulation and febrinil 650 was procure from commercial pharmacist shop standard working solution.

Standard stock solution of paracetamol was prepared by dissolving accurately weighed 10 mg of drug in 10 mL of mobile phase (actonitrile and water 60 : 40 v/v) HPLC grade and filter through 0.25 µ membrane as external standard.

Sample solution

Twenty tablet of Febrinil each containing 650 mg of paracetamol weighted and finely powdered in mortar. A quantity equivalent to 50 mg of paracetamol weighed and transferred to a volumetric flask and dissolved in 50 mL of mobile phase i.e. mixture of acetonitrile and water (60 : 40 V/V.) This sample solution was stirred magnetically for five min. and centrifuged at 1000 rpm. It was diluted to get the solution of 0.5 mg/mL (500 µg) and 0.05 mg (50 µg) concentration filter through 0.25 µ membrane and degassed.

RESULTS AND DISCUSSION

Method development

HPLC analysis was performed by isocratic elution. The mobile phase was selected after several trials with acetonitrile and water maximum detection and sensitivity was observed in mobile phase ratio 60 : 40 v/v/ (ACN and Water). All the solvent were filtered through 0.45 µ Millipore filler before used and degassed in an ultrasonic bath. Standard sample solutions were also filtered through 0.25 µ membrane and degassed. 20 µL of standard solution was injected to Rhenodyne injector several time to optimized the condition. A steady baseline was recorded at the flow rate 1 mL/ min at the retention time 2.690 minute and UV detection at 210 nm. A typical chromatogram of paracetamol is given in Fig. 1 a and b. Throughout the study, the suitability of the chromatograph is system was monitored by calculating the resolution, selectivity and peak symmetry. Optimized chromatographic conditions are listed in Table 1.

Table 1:

Parameter	Optimized condition
Chromatograph	Shimadzu HPLC
Column	Phenomenex C_{18} Column (250 mm x 4.6 mm 5 µ)
Mobile phase	Acetonitrile and water (60 : 40 V/V)

Cont…

Parameter	Optimized condition
Flow rate	1.0 mL/min
Detection	UV at 210 nm
Injection volume	20 µL
Temperature	Ambient
Retention time of paracetamol	2.690 min

Method was validated with respect to precision, accuracy stability, specificity, linearity, LOD, LOQ ruggedness and robustness according to ICH guidelines.

Accuracy

Accuracy of the method was determined by recovery experiments. The prepared standard solution was injected six times as a test sample from the respective area counts the concentration of the of paracetamol was calculated using the detector responses. The precision of the method was demonstrated by interday and intraday variation studies the response factor of the drug peak and % RSD were calculated. From the data developed HPLC method was found to be precise.

Linearity

The Linearity of the method was determined at seven concentration levels ranging from 20-100 mg/ mL. The calibration curve was constructed by plotting response factor against concentration of drugs. The slope and intercept value for calibration curve was Y = 596.20 x + 12. 8458 (Table 3) shows the regression equation correlation coefficient (r^2) RSD value of the slope, intercept LOD and LOQ values.

The limit of detecting LOD and limit of quantification of (LOQ) the developed method by injecting progressively low concentration of std solution using the developed RP HPLC method. The LOD of drug was found to be 0.02 µg / mL and LOQ was 19.29 µg /mL. The ruggedness of the method was tested by injecting the standard working solution into different days. The high degree of reproductivity of detector responses and retention times indicate that the method is fairly rugged.

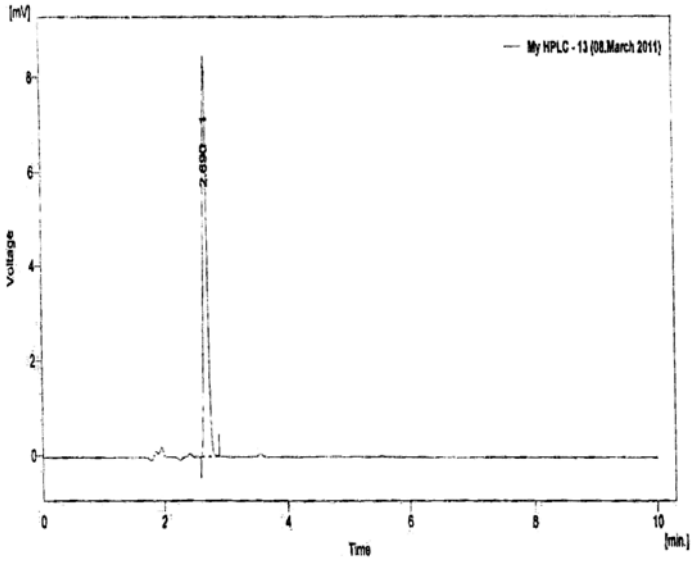

Fig. 1(a): Standard chromatograph

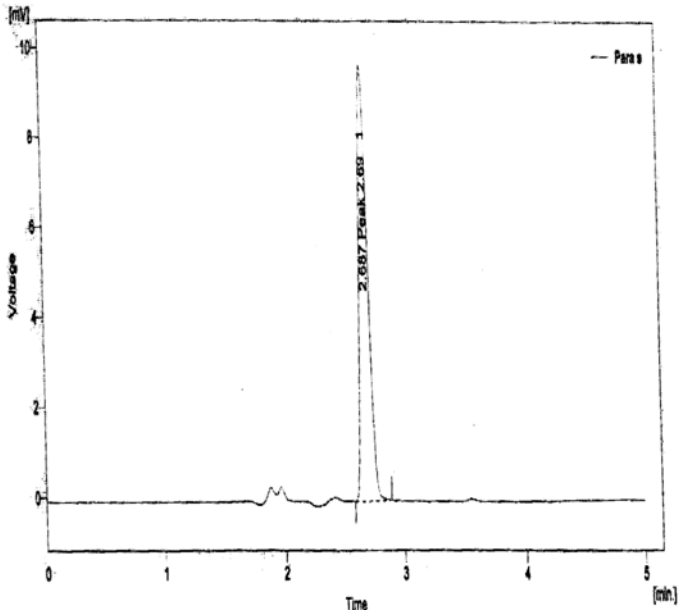

Fig. 1(b): Sample chromatograph

The stability of standard and sample solutions were evaluated under different storage condition for short term stability, solutions were kept of room temperature for 24 hrs. The long terms stability was assessed after storage of stock solution at 4^{o}C for 15 days. The results were evaluated by comparing peak area ratio for standard solutions and sample solutions with those of freshly prepared solutions. The result found within 90-91 % of initial values indicates the stability of solution.

The method developed was found to be specific for the quantitative determination of paracetamol bulk drugs like febrinil Sample solution was analyzed in triplicate after preparation of the drug solution as mentioned above is experimental section. The amount of paracetamol was found to be within the range of 90-91 % none of the excipients were found to interfere with the analyte peak and the results.

CONCLUSION

The developed method is suitable for the identification and quantification of the paracetamol in single dosage from A. high percentage of recovery shows that the method can be successfully used on a routine basis.

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