



DEVELOPMENT AND VALIDATION OF RP – HPLC METHOD FOR THE ESTIMATION OF CEFETAMET IN BULK AND TABLET DOSAGE FORM

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ABSTRACT

A simple, rapid, sensitive and precise High Performance Liquid Chromatography (HPLC) method has been developed for the estimation of cefetamet in bulk and tablet dosage form. In this method, RP-C₁₈ column (150 mm x 4.6 mm I.D., 5 µm particle size) with mobile phase consisting of acetonitrile, methanol and phosphate buffer (pH 3.60) in the ratio of 50 : 20 : 30 v/v/v in isocratic mode was used. The detection wavelength is 236 nm and the flow rate was 0.8 mL/min. In the range of 5-25 µg/mL, the linearity of cefetamet shows a correlation coefficient of 0.9995. The proposed method was validated by determining sensitivity, accuracy, precision and system suitability parameters. The method is simple, fast, accurate and precise and hence, it can be applied for routine analysis of cefetamet in bulk drug and its tablet dosage form.

Key words: RP-HPLC, Cefetamet, Validation.

INTRODUCTION

Cefetamet is a third generation cephalosporin antibiotic. It is given orally as cefetamet pivoxil hydrochloride. Cefetamet was effective in the treatment of otitis media, pneumonia, uncomplicated gonorrhoea, pharyngotonsillitis, urinary tract, upper and lower respiratory tract infections^{1,2}. Cefetamet is chemically 7-[2-(2-aminothiazol-4-yl)-2-methoxy imino acetamido]-3-methyl 3-cephem-4-carboxylic acid. It is not official in any pharmacopoeia. The literature survey reveals that few analytical methods were established for the quantitative estimation of cefetamet in bulk drug and pharmaceutical dosage forms³⁻⁹.

The present investigation reports a simple HPLC method for the analysis of

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cefetamet in bulk as well as in tablet dosage form.

EXPERIMENTAL

The separation was carried out on HPLC (Waters) system with Waters 1525 binary HPLC pump, Waters 2487 UV dual λ absorbance detector, Waters Breeze software and RP-C₁₈ column. The mobile phase consisting of acetonitrile, methanol and phosphate buffer (pH 3.60) in the ratio of 50 : 20 : 30 v/v/v in isocratic mode was used. The detection wavelength is 236 nm and the flow rate was 0.8 mL/min. The detection was monitored at 236 nm and the run time was 6 min.

The standard solution of cefetamet (1 mg/mL) was diluted to get 5 different concentrations ranging from 5-25 μ g/mL. The solutions were injected into the 20 μ L loop and the chromatograms were recorded. The calibration graph was constructed by plotting concentration v/s peak area ratio. The linearity experiment was carried out in triplicate to ascertain accuracy and precision of the method.

Assay

Twenty tablets were weighed and powdered. A quantity of powder equivalent to 100 mg of cefetamet was weighed accurately, transferred to 100 mL calibrated volumetric flask, dissolved in distilled methanol and made up to 100 mL with methanol. From this solution, further dilutions were made in mobile phase to get the appropriate concentrations. These solutions were injected and the chromatogram was recorded. The amount of cefetamet was determined from the regression equation. The results were given in Table 1.

Table 1. Assay and recovery studies

Formulation	Label claim (mg)	% of amount found*	C.V. (%)	Amount of standard added (mg)	Amount recovered*	% recovery
Brand-1	250	98.2	0.22	100	99.6	100.2
Brand-2	250	101.6	0.20	100	100.19	101.37

*Mean of five determinations

Validation of proposed method

Selectivity of the method was assessed on the basis of elution of cefetamet using the above mentioned chromatographic conditions. Precision was ascertained by the

determination of intra-day and inter-day variabilities. To study the accuracy, reproducibility and precision of the proposed method, recovery experiments were carried out in triplicate by adding a known amount of drug to preanalysed sample and the percentage recovery was calculated. The results are reported in Table 2.

Table 2. Validation summary

System suitability	Results
Theoretical plates (N)	8129
Linearity range ($\mu\text{g/mL}$)	5-25
Percentage recovery (Accuracy)	98.68
LOD ($\mu\text{g/mL}$)	0.025
LOQ ($\mu\text{g/mL}$)	0.05
Tailing factor	1.071
Capacity factor	4.786
Symmetry factor	1.074

RESULTS AND DISCUSSION

By applying the proposed method, the retention time of cefetamet was found to be 3.4 min. (Fig. 1.).

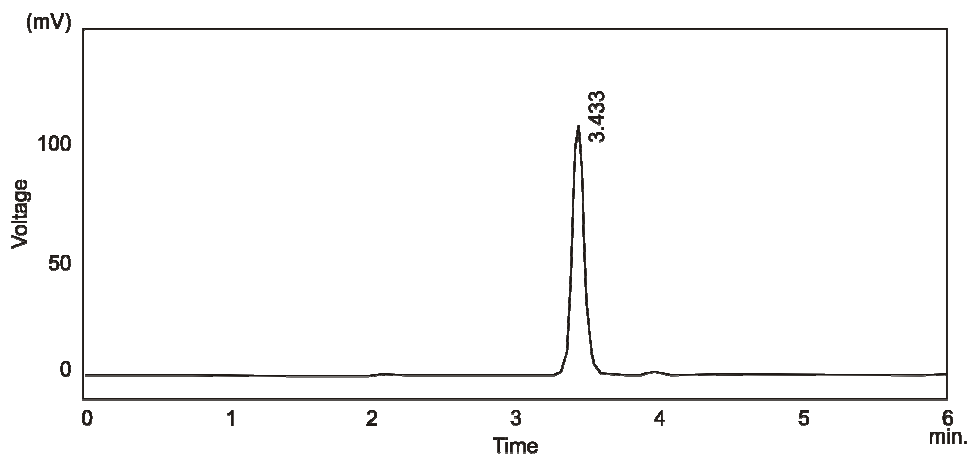


Fig. 1: A typical chromatogram of cefetamet

Linearity was obeyed in the concentration range of 5-25 µg/mL. The regression equation of cefetamet concentration over its peak area ratio was found to be $Y = -2.03 + 25.631 X$ ($r = 0.9995$) where X is the concentration of cefetamet (µg/mL) and Y is the peak area ratio. The high percentage of recovery indicates that the proposed method is highly accurate.

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