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Gas chromatographic determination of celecoxib from tablet

U.M.Talekar, S.J.Kokate, H.R.Aher, S.R.Kuchekar*

P. G. Department of Analytical Chemistry, P. V. P. College, Pravaranagar, At/Po. Loni (Kd), Tal. Rahata, Dist. Ahmednagar, MS, 413713, (INDIA)

E-mail : shashi17@gmail.com

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ABSTRACT

A gas chromatographic technique was developed for the quantitative determination of non-steroidal anti-inflammatory drug (NSAID) celecoxib in its solid dosage form. p-Nitro aniline is used as internal standard. In proposed method HP-5 capillary column (15 meter length, 0.53 mm i.d., 1.5 u film thickness), nitrogen as carrier gas with flow rate 12 psi., oven temperature programming with split type injector was used and detector is a flame ionization detector. The retention time of celecoxib and p-Nitro aniline is 4.6 min and 1.5 min respectively. Linearity is in the range of 2 mg/ml to 7 mg/ml (r^2=0.9999). The percentage recovery is 99.8%. The proposed method is useful for quantitative estimation of celecoxib in its tablet form. It is simple, fast, accurate and precise.

KEYWORDS

Gas Chromatography;
Celecoxib tablet.

INTRODUCTION

Celecoxib is non-steroidal ant-inflammatory drug (NSAID). Chemically it is 4-[5-(4-methyl phenyl)-3(trifluromethyl)-1H Pyrazol -1-yl] Benzene sulfonamide. It is used to treat arthritis, pain, menstrual crams and clonic polyps. Literature survey reveals that this drug is not in any pharmacopoeia. Celecoxib is determined from blood, after treating blood serum with tolbulamide, extracted in dichloromethane and extract is evaporated under nitrogen. Residue left is dissolved in mobile phase and celecoxib is determined by HPLC[1]. Clecoxib from human plasma was determined by using HPLC[2] based on liquid-liquid extraction with chloroform and reversed phase chromatography. Carboxy celecoxib and hydroxyl celecoxib in human plasma separated by HPLC[3] and determined by spectrophotometric method .HPTLC[4] was developed for determination of celecoxib from its dosage form. It is determined from capsule using HPLC[5] after separation on C18 reversed phase column. HPLC[6] was used for determination of celecoxib from human plasma using solid phase extraction. Revered phase HPLC[7] was developed for separation and determination of processes related impurities of celecoxib. It is detected from human plasma by using HPLC[8] Method was developed for simultaneous determination of two COX-2 inhibitors, celecoxib and rofecoxib in addition to sodium diclophenac and niffumic acid in human serum[9]. Among these methods limited methods used for estimation of celecoxib from capsule formulations while maximum methods give estimation of this drug from human plasma. There is not single method available for estimation of celecoxib from formulated tablets by using gas chromatography; hence, the development of gas chromatographic method for celecoxib from pharmaceutical formulations is an analytical merit.

Recently in our laboratory method was developed

for quantitative determination of ambroxol hydrochloride[10] [4 (2-amino-3, 5-dibromophenyl) methyl amino cyclohexanol hydrochloride] from formulated formulation by gas chromatography. In extension to our previous work, a gas chromatographic technique was developed for the quantitative determination of non-steroidal anti-inflammatory drug (NSAID) celecoxib in its solid dosage form. The percentage recovery is 99.8%. The proposed method is useful for quantitative estimation of celecoxib in its tablet form. It is simple, fast, accurate and precise.

EXPERIMENTAL

Reagents: Dichloromethane, anhydrous sodium sulphate, p-nitro aniline and methanol supplied by Unichem Laboratories India Ltd. Dematerialized water used from two-bed portable deionizer make WTC model No CA 7'. Perkin Elmer make model Clarus 500 Gas Chromatographic equipment was used to carry out proposed work.

Sample and Standard Solutions

Celecoxib (Pure reference standard) Solution A: Weigh accurately 2500 mg of pure reference standard of celecoxib (purity 99.8%) and transferred it in a beaker, dissolve in dichloromethane. Transfer this solution to 50 ml volumetric flask and diluted it up to the mark using dichloromethane.

(Internal Standard) Solution B: Transfer accurately weighed 2500 mg p-nitroaniline in a beaker, dissolve it in methanol, then transfer solution to 50 ml of volumetric flask and diluted it up to mark using methanol.

Reference Standard Solution: Transfer 2.5 ml of solution A and 2.0 ml of solution B to 25 ml of volumetric flask and diluted it up to the mark with dichloromethane.

Sample Solution: Weigh and powder 10 celecoxib tablets (200 mg celecoxib per tablet). Powder equivalent 250 mg celecoxib was taken in to separating funnel containing 10 ml of water. This solution was extracted four times with dichloromethane. After separation of layers, organic layer has dried over anhydrous sodium sulphate and dried solution was transferred to 50 ml volumetric flask. To this solution 4 ml solution B is added and diluted up to mark using dichloromethane.

Working Standard Solution: Aliquots of standard solution was transferred in 25 ml volumetric flask such a that final concentration is in a range 2 to 7 mg/ml, in which 2 ml internal standard solution B was added and diluted up to mark with dichloromethane.

Method: Peak area ratio was recorded for celecoxib and p-Nitroaniline to study the accuracy, reproducibility and precision of the proposed method. Recovery experiments were carried out with fixed amount of preanalyzed sample and standard stock solution at three different levels. Each level had repeated three times. The separation was carried out on Perkin Elmer gas chromatograph equipped with split injection port and flame ionization detector.

Chromatographic conditions

Column	: HP-5 (15mtrx0.53 mx1.5u);
Carrier pressure	: 12psi;
Detector Temperature	: 280°C;
Split Ratio	: 10:1;
Carrier gas	: Nitrogen;
Injector Temperature	: 280°C;
Attenuation	: 8;
Injection volume	: 1ul, Range: 1;
Oven Temperature	: 150°C (1min) @40°C/min to 280°C (5min).

RESULTS AND DISCUSSION

The assay results from four different batches of celecoxib by gas chromatography was reported in TABLE 1. These results are closely related to claimed amount of celecoxib indicating that, the proposed method is precise and accurate. The mean recovery of celecoxib is 99.8% (TABLE 2) indicates that in a method no interference from excipients. The plot of peak area ratio versus the respective concentration (Figure 1) was found to be linear over the range of 2 to 7 mg/ml with coefficient of regression (r^2=0.9999). The proposed method gave good resolution between celecoxib and p-nitroaniline (Figure 2). It is simple, rapid, and precise therefore rec-

TABLE 1 : Assay results

Amount claimed mg/tablet	Amount found mg/tablet
200	199.24
200	198.92
200	198.60
200	199.30
200	199.38

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ommended for routine quality control analysis of celecoxib.

TABLE 2 : Recovery study for spiked concentrations of celecoxib

Level	Label claim mg/tablet	Amount found mg/tablet	% Recovery	Mean Recovery
Level 1	200	200.64	100.3	
Level 2	200	199.32	99.1	99.8%
Level 3	200	200.04	100.0	

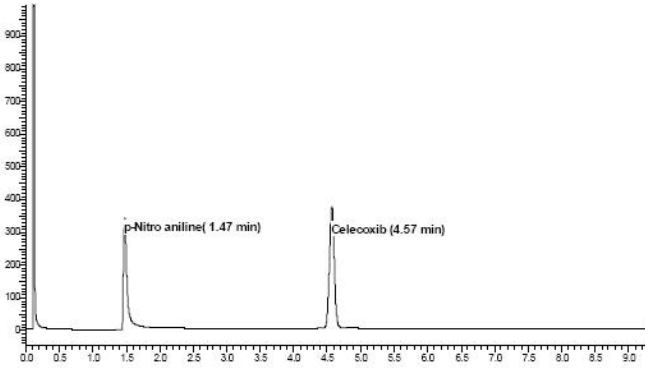

Figure 1 : Typical chromatogram of standard solution of celecoxib and P-nitro aniline

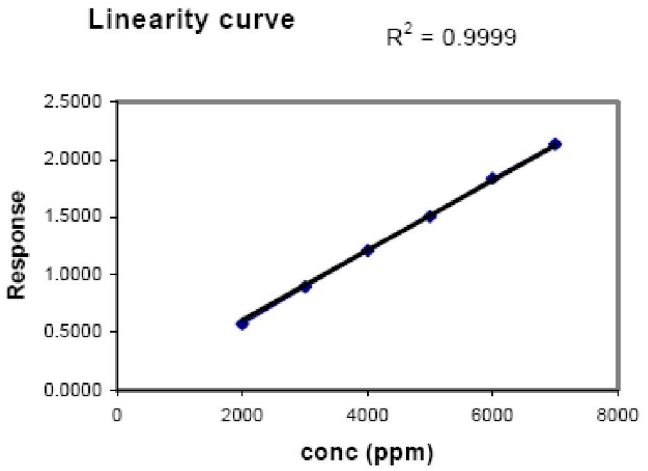

Figure 2 : Linearity graph

CONCLUSION

Proposed method is verified by analyzing various concentrations of standard having range 7 to 12 mg/ml, gave linearity. It is simple, accurate and precise method. It useful in routine analysis in quality control departments at pharmaceutical industries.

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