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HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC ASSAY FOR THE DETERMINATION OF PARACEFAMOI, PSEUDOEPHEDRINE HCI AND TRIPROLIDINE HO IN PHARMACEUTICAL PREPARATIONS.

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ABSTRACT

The knowledge of UV. absorption characteristics of paracetamol, pseudoephedrine HCI and tripmlidine HCI has been used to determine these compounds by HPLC method in pharmaceutical preparations. The method involves reverie phase chromatography with isocratic mode of elution. Rather selecting a compromised wavelength for all the three components, wavelength switching' programme has been executed to give reasonable UV responses because of the widely different W characteristics of these components. This method performs well in term of the precision and accuracy as indicated by linear regression analysis. The RSD of the method is 1.12 to 187% and the recovery is 103.10- 9556%.

INTRODUCTION

Paracetamol, pseudoephedrine HCI and triprolidine HCI are classified as basic drugs. Most of the basic drugs have been analysed using 125mm spherisorb S5w silica column and methanolic ammonium perchlorate (10 mM, pH 6.7) as eluent using electrochemical mode of detection (1). Although UV absorption and fluorescence detection have been widely used in the analysis of basic drugs, oxidative mode of electrochemical detection (2) has been restricted to compounds containing relatively easily oxidised groups such as phenolic hydroxyl or phenothiazine sulphur. There are a number of pharmaceutical preparations containing a mixture of paracetamol, pseudoephedrine HCI and triprolidine HO as active componentts. Widely different UV characteristics of all the three components can be judged from the specific absorption value at different wavelength. The absorbance (1%, 1cm) of a solution of paracetamol at 249nm is about 80, while for triprolidine HCI the absorbance at 290 nm is about 1.2. The light absorption of pseudoephedrine HCI in the range 230 to 350 nm of a 0.05%, w/v solution exhibits three maxima, at 251, 257 and 263 ram. The absorbance at 251 nm is about 0.75, at 257 nm, about 0.98 and at 263 nm about 0.78 (4).

Quantitative analysis of this mixture normally requires two separate isocratic high performance liquid chromatographic (HPLC) systems because of the widely differing amounts normally present in this type of products and different UV characteristics of the three components.

A reverse phase chromatographic method using 250x5mm partisil 10 ODS column, an eluent based on alcohol and ammonium acetate solution (3) and UV detection with the "wavelength" switching programme, has been reported to quantify all the three components simultaneously. The object of this study is to perform analysis with a single HPLC method rather than two separate HPLC determinations *for a* pharmaceutical preparation.

EXPERIMENTAL

Materials

Ammonium acetate GR, and ethanol were obtained from Merk. All stock solutions were prepared by dissolving USP reference standards of paracetamol, pseudoephedrine HCI and triprolidine HCI.

HPLC Method

The chromatographic set up used in this study included a LC-6A pump (Shimadzu, Japan) for delivering the ducat; SIL-6A injector; SPD-6AV detector; integrator C-R3A and partisil 10 ODS 250x5mm column protected with pre-column ODS.

A suitable degassed and filtered mixture of alcohol and 0.11% aqueous acetate solution (700:300) has been used as eluent. The flow rate was maintained at 1.51ml/min. Detection was performed at 300nm followed by a wavelength switching programme entered in lime programme" mode of system controller SCL-6A module (Shimadzu) which provides a "built-in" facility to change the parameters like wavelength, flow rate etc. automatically during an analysis. By selecting from the menu box appeared on the screen in 'Time program" mode, enter the requ[i]red parameter which is to be changed after certain period of time. The method presented, required a change of wavelength from 300 nm to 257 nm after 5.6 minutes and to stop after 16 minutes of run time. All separations *were* carried out at ambient temperature.

Preparation of Authentic standard Solution

A 150mg quantity of pseudoephedrine HCI (USP reference standard) was transferred into a 100m l volumetric flask. It was dissolved in and diluted to volume with a mixture of ethanol and water (1:1, v/v), and mixed well. This was designated as solution "A".

A 125 mg quantity of triprolidine NCI (USP reference standard) was placed into a 200 nil volumetric flask. The substance was dissolved in and diluted to volume with a mixture of alcohol and water (1:1, v/v), and mixed well. This was designated as solution "B".

A 125 mg quantity of paracetamol (USP reference standard) was transferred into a 250ml volumetric flask. The contents were dissolved in 100 ml of a mixture of ethanol and water (1:1, v/v).

A 10 ml portion of solution "A" and 1 ml of solution "B" were transferred into the 250 ml volumetric flask containing paracetamol. The contents of the flask were diluted to volume with a mixture of ethanol and water (1:1, Wv) and mixed well. This is standard solution. The solution was filtered through 0.45 um membrance filter before injecting to L.C.

Preparation of Test Solution

A test solution of the following concentrations of paracetamol, pseudoephedrine HCI and triprolidine HCI using a mixture of ethanol and water (1:1, v/v) as diluent was prepared.

Paracetamol	500 µm/ml
Pseudoephedrine HCl	60 µg/ml
Triprolidine HCl	2.5 µg/ml

The test solution was filtered through 0.45 pm membrane filter before injecting to L.C.

Approximate Retention Time (Minute)

The approximate retention times of various compounds were measured as follows:

Paracetamol	2.5 min
Pseudoephedrine HCl	6.7 min
Triprolidine HCl	12.5 min

RESULT AND DISCUSSION

Individual test and standard solutions of 40,80,100, 120 and 140% of the theoretical concentration of each of the active ingredients were examined by HPLC method and the[i]r UV responses were measured. In both cases, i.e. the test and HPLC of paracetamol 32 standard, a linear behaviour was observed when a graph is plotted between peak height and concentration.

Regression analysis of the data for each component was carried out and the values of slope, intercept and correlation coefficient for each calibration curve are given in Table 1.

Table 1

Regression analysis of calibration curves for paracetamol, pseudoephedrine HCl, and triprolidine HCl.

Drug	Slope	Intercept	Correlation Coefficient
Paracetamol	2.23	39.77	0.99892
Pseudoephedrine HCl	26.55	2.06	0.998896
Triprolidine HCl	11.83	-11.10	0.999697

The validity of the listed regression data was tested by the assay of the authentic mixture containing known quantities of paracetamol, pseudoephedrine HCI and triprolidine HCL The result showed good accuracy indicated by the percentage recovery (Table 2).

The method is specific, sensitive, and time saving and can be used in the quality control and R&D Laboratories for similar type and composition of products.

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