

Short communication

# Capillary gas chromatographic determination of phenylpropanolamine in pharmaceutical preparation

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## Abstract

Analytical procedure has been developed for the gas chromatographic determination of phenylpropanolamine (PPA) using trifluoroacetylacetone (FAA) as derivatizing reagent. Elution is carried out from the column HP-5 (30 m × 0.32 mm i.d.) with film thickness 0.25 μm at initial column temperature 70 °C for 5 min, followed by heating rate 10 °C/min up to 120 °C. Injection port temperature was maintained at 270 °C. Nitrogen flow rate was 2 ml/min and detection was by FID.

The linear calibration curve was obtained with 30–150 μg/ml PPA with detection limit of 6.0 μg/ml. The method was used for the determination of PPA from Sinutab and Tavegyl-D tablets. The relative standard deviation (R.S.D.) for the analysis of pharmaceutical preparation was obtained within 0.4–0.9%.

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**Keywords:** Phenylpropanolamine; Trifluoroacetylacetone; Pharmaceutical

## 1. Introduction

Phenylpropanolamine hydrochloride (PPAx·HCl) is a sympathomimetic agent with vasoconstrictor and decongestant effects on inflamed mucous membranes. It is also reported as an appetite suppressant. It is used in over-the counter (OTC) and prescription medications for cough and cold. Recently, considerable interest in PPA has increased due to the serious side effects accompanied its use including hemorrhagic stroke, arrhythmias and hypertension [1–5]. This caused Federal Drug Administration (FDA) of United States to ask OTC manufactures to reformulate products containing PPA to remove PPA from the market and issue public advisory warning about the risks linked to PPA [6,7].

A number of analytical methods have been reported for the determination of PPA mostly based on spectrophotometry [8,9], spectrofluorimetry [10], room temperature phosphorescence [11], fluoroimmunoassay [12], radioenzymic assay [13], Raman spectroscopy [14], capillary electrophoresis [15,16], thin layer [17], gas (GC) [18–24] and liquid chromatogra-

phy (LC) [25–34]. The procedures based on GC and LC have been more frequently reported. The GC methods are based on precolumn derivatization with *N*-methyl-*N*-trimethylsilyl-trifluoroacetamide [22], *N*-methyl-bis-(heptafluorobutyramide) [35,36], heptafluorobutyric anhydride [35] and cyclohexane [35]. However, some column damage problems have been reported using acetylation reagents. The simple β-diketone could react with primary amino compound to form highly stable Schiff base and enhance the sensitivity of FID detection due to increase in carbon chain length. Trifluoroacetylacetone (FAA) has been used for the GC determination of diamino-compounds putresine and cadaverine from biological fluids [37], but the present study examines FAA for the GC determination of PPA.

## 2. Experimental

### 2.1. Materials

Trifluoroacetylacetone (Fluka), phenylpropanolamine hydrochloride (norephedrine hydrochloride) (Sandoz Pak.), ephedrine hydrochloride [(1-methylaminoethyl) benzyl alcohol hydrochloride] (Merck), methanol (E. Merck), chloroform

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(E. Merck), sodium hydroxide (Fluka), hydrochloric acid (37%), potassium chloride, acetic acid, sodium acetate, ammonium chloride and ammonia (E. Merck) were used. Buffer solutions at unit pH interval were prepared from the following: hydrochloric acid (1.0 M), potassium chloride (1.0 M), acetic acid (1.0 M), sodium acetate (1.0 M), ammonium acetate (1.0 M), ammonia (1.0 M), ammonium chloride (1.0 M), sodium carbonate (saturated) and sodium bicarbonate (1.0).

## 2.2. Instrumentation

Spectrophotometric studies were carried out using a double beam Hitachi 220 spectrophotometer (Hitachi (PVT) Ltd., Tokyo) with dual 1 cm silica cuvettes. pH measurement were made with Orion 420 A pH meter with glass electrode and internal reference electrode (Orion Research Inc., Boston, USA).

The gas chromatographic studies were carried out on Agilent model 6890 Net work GC system gas chromatograph (Agilent Technologies Inc., USA) coupled with flame ionization detection (FID), hydrogen generator Parker-Balson model H2-90, Analytical gas system (Parker Hannifin Haverhill, MA, USA) and pure nitrogen (British Oxygen Company (BOC), Karachi). The gas chromatograph was controlled by the computer with Chemstation software (Agilent Technologies). Capillary GC column HP-5 (30 m × 0.32 mm i.d.) with film thickness 0.25 μm (J & W Scientific GC column, USA) was used throughout the study.

## 2.3. GC–FID method

To the solution of phenylpropanolamine hydrochloride (0.2–1 ml) containing (30–150 μg) was added sodium hydroxide (0.5 ml, 0.2% w/v) in water and chloroform (2 ml). The contents were mixed well and layer was allowed to separate. Exactly 1 ml of chloroform layer was transferred to screw cap vial and was added FAA (1 ml, 1% v/v) in methanol and acetic acid (0.1 ml). The contents were heated at 75 °C for 10 min and the residue was dissolved in methanol (0.5 ml). The solution (1 μl) was injected on the column HP-5 (30 m × 0.32 mm i.d.) with film thickness 0.25 μm at column temperature 70 °C for 5 min, followed by heating rate 10 °C/min up to 120 °C with total run time 20 min. Injection port and detector temperatures were maintained at 270 and 300 °C. Nitrogen flow rate was 2 ml/min with split ratio 10:1. FID was used for detection.

## 2.4. Analysis of phenylpropanolamine from pharmaceutical preparation

Ten tables each Tavegyl-D (Sandoz Pak. Ltd., Karachi) containing 75 mg/tablet and Sinutab (Parker-Davis and Co., Pak. Ltd., Karachi) were well ground. Tavegyl-D 0.512 g and Sinutab 0.475 g in triplicate were dissolved in water separately. The solution was filtered and volume was adjusted to 10 ml. The solution (0.1 ml) for Tavegyl-D and (0.2 ml) for Sinutab were taken and processed as above. The amounts of drug from pharmaceutical preparations were calculated using external calibration curve.

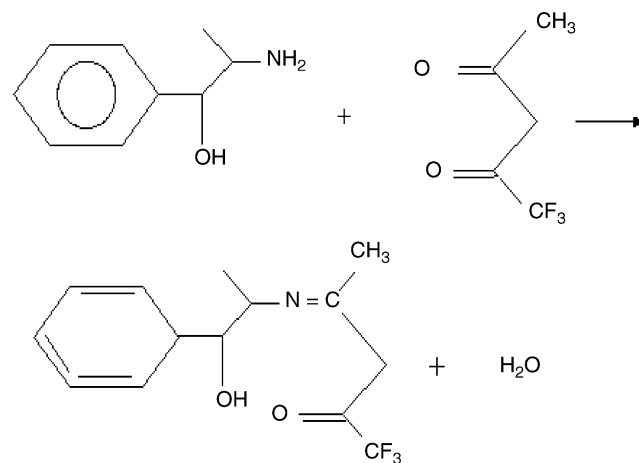


Fig. 1. Schematic reaction of PPA with FAA.

## 3. Results and discussion

Acetylacetone and FAA were examined for GC determination of PPA, but FAA gave better sensitivity due to the presence of trifluoromethyl group in FAA, which could enhance the thermal stability and volatility of derivative. The derivatizing reagent FAA was, therefore, selected.

PPA derivative formed with FAA (Fig. 1) absorbed at 323 nm with molar absorptivity of 11,211 l/mol cm and supported the formation of derivative. Any change in absorbance up to 24 h was not observed. After extraction of amine in chloroform from alkaline solution, the derivatization was carried out in chloroform-methanolic media, in the presence of acetic acid. The effect of pH, the concentration of derivatizing reagent and heating time on the derivatization was examined. Each time absorbance of the solution was measured against reagent at 323 nm and the condition, which gave maximum response, was considered as optimum. The effect of pH was examined within pH 1–10 and a similar response was obtained within pH 1–3.5 and addition of acetic acid covered the pH range satisfactory (Fig. 2).

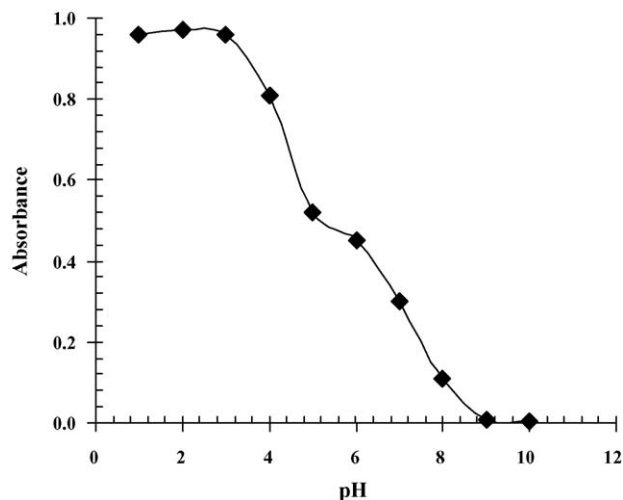


Fig. 2. Effect of pH on derivatization of PPA with trifluoroacetylacetone.

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