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Spectrofluorimetric analysis of famotidine in pharmaceutical preparations and biological fluids by derivatization with benzoin

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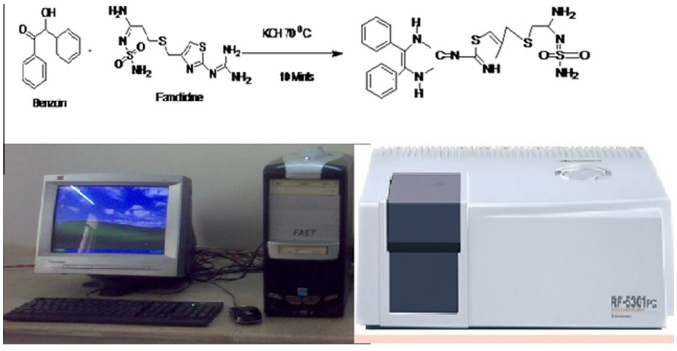
HIGHLIGHTS

- Article is based on derivatization of famotidine and rapid method development by using spectrofluorimeter.
- Pharmaceutical products have been analyzed by spiked and standard addition methods.
- Blood and urine samples also have been analyzed by spiked procedures.

GRAPHICAL ABSTRACT



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ABSTRACT

A sensitive and simple spectrofluorimetric method has been developed for the analysis of famotidine, from pharmaceutical preparations and biological fluids after derivatization with benzoin. The reaction was carried out in alkaline medium with measurement of fluorescence intensity at 446 nm with excitation wavelength at 286 nm. Linear calibration was obtained with 0.5–15 μg/ml with coefficient of determination (r^2) 0.997. The factors affecting the fluorescence intensity were optimized. The pharmaceutical additives and amino acid did not interfere in the determination. The mean percentage recovery ($n = 4$) calculated by standard addition from pharmaceutical preparation was 94.8–98.2% with relative standard deviation (RSD) 1.56–3.34% and recovery from deproteinized spiked serum and urine of healthy volunteers was 98.6–98.9% and 98.0–98.4% with RSD 0.34–0.84% and 0.29–0.87% respectively.

Introduction

Famotidine, (3-(((2-((aminoiminomethyl) amino)-4-thiazoyl)-methyl) thio)-N-(aminosulfonyl) propanimidamide) (Fig. 1) is a potent competitive and reversible inhibitor of histamine action at H_2 receptors and is used for the treatment of active duodenal ulcer, gastric ulcer, heartburn, acid indigestion, sour stomach and Zollinger-Ellison syndrome [1,2]. The famotidine is commonly given as 40 mg daily as therapeutic doze to the patients [3].

The determination of famotidine in pharmaceutical preparations and biological fluids has attracted a considerable attention and a number of procedures have been developed mainly based on spectrophotometry [4–8], potentiometry [9], polarography [10], spectrofluorimetry [11–14], high performance liquid chromatography (HPLC) [15–19], flow injection [20,21], capillary zone electrophoresis [22] and gas chromatography [23].

The chromatographic and electrophoretic techniques have the capability for simultaneous determination of multicomponent

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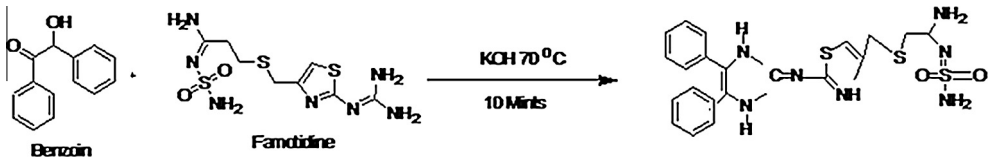

Fig. 1. Reaction between benzoin and famotidine in alkaline solution.

system, but for the analysis of single component, spectrofluorimetric procedure gives adequate sensitivity and selectivity with the aid of a suitable derivatizing reagent. The spectrofluorimetric procedures for the determination of famotidine are based on the use of derivatizing reagents 1,4-benzoquinone [11], 9.10-phenanthraquinone [13] and through ternary complex formation with some lanthanide ions [14]. Recently benzoin has been used for the spectrophotometric determination of famotidine [24] and the present work reports the development of sensitive spectrofluorimetric method for the analysis of famotidine from pharmaceutical preparations and biological fluids (serum and urine) after derivatization with benzoin.

Experimental

All the chemicals used were of reagent grade. The stock solution of famotidine (Sigma, St. Louis, MO, USA) containing 1 mg/ml was prepared by dissolving 100 mg of the drug in of in methanol (30 ml) and volume was adjusted to 100 ml with deionized distilled water. Buffer solution (0.1 M) between pH 1 and pH 11 at 0.5 unit interval was prepared from the following; potassium chloride adjusted with hydrochloric acid (pH 1–2), acetic acid–sodium acetate (pH 3–6), ammonium acetate (pH 7), boric acid–sodium tetraborate (pH 7.5–8.5), sodium bicarbonate–sodium carbonate (pH 9), ammonium chloride–ammonia solution (pH 10), and sodium chloride–sodium hydroxide (pH 11). Benzoin (Fluka, Buchs, Switzerland) solution (4.0 mmol/L) was prepared by dissolving 85 mg benzoin in 100 ml of methanol.

Equipments

The pH measurements were taken with an Orian 420 A pH meter (Orion Research Inc. Boston, USA) with combined glass electrode and reference internal electrode. Shimadzu Model RF-5301 PC, spectrofluorometer (Shimadzu, Kyoto, Japan) was used. The spectrofluorometer was controlled by the computer with RFPC software. Quartz cuvette with 1 cm pathway was used for fluorescence measurements.

Analytical procedure

The solution 0.5–2.0 ml containing 5–150 μg famotidine was transferred to 10 ml volumetric flasks and was added to 0.5 ml of 2 M potassium hydroxide, 0.5 ml of benzoin solution (4.0 mmole/L in methanol), 0.5 ml of 2-mercaptoethanol (8 mmol), and 0.4 ml, 0.2 mol/L sodium thiosulphate. The content was warmed in water bath for 10 min at 70 °C and contents were allowed to cool. The solution was added to borate buffer 0.5 ml, pH 8.5 and the volume was adjusted to the mark with distilled water. The fluorescence intensity of the resulting solution was measured at 446 nm excitation at 286 nm (Fig. 2). The blank determination without the addition of the analyte was also prepared simultaneously and was used to adjust the background fluorescence intensity.

Analysis of pharmaceutical preparations

Five tablets of each Ulfam (Focus & Rulz Pharmaceuticals Pvt. Ltd., Islamabad, Pakistan), Femme (Batala Pharmaceuticals, Gujranwala, Pakistan), Famotin (Efroze, Korangi Industrial Area, Karachi, Pakistan) and Facid (Matador Chemicals, Lahore, Pakistan) with labeled amount of 40 mg/tablet were weighed and crushed to fine powder. The powder corresponding to one tablet was weighed and dissolved in methanol–water (1:1 v/v). The solution was filtered and volume was adjusted to 100 ml with methanol water (1:1 v/v). The solution (1 ml) was further diluted to 10 ml and solution (1 ml) was transferred to 10 ml volumetric flasks and the analytical procedure was followed. The quantitation was made from the external calibration using linear regression equation based on $y = ax + b$.

Analysis of pharmaceutical preparations from spiked samples

Five tablets of each pharmaceutical preparation were treated as analysis of pharmaceutical preparation. The solutions (0.5 ml) in duplicate were taken from the final solution prepared and a solution was added to famotidine to adjust final concentration of 2.5 μg/ml and both the solutions were processed as analytical procedure. The quantitation was made from the linear calibration curve and an increase in the response with added standards.

Analysis of famotidine from spiked serum samples

The blood sample (5 ml) was collected from healthy volunteers who have not taken any medicine at least one preceding week. The blood sample was allowed at room temperature for 1 h and centrifuged at 3000*g* for 20 min. The supernatant layer was collected and added to 5 ml of methanol. The mixed contents were again centrifuged at 3000*g* for 15 min. The clear supernatant layer was collected. Three aliquots of 1 ml each were taken and aliquots 1 and 2 were added to famotidine to adjust final concentration of 3.50 μg/ml and 5.50 μg/ml respectively. All the three solutions were processed as analytical procedure. Aliquot 3 was treated as

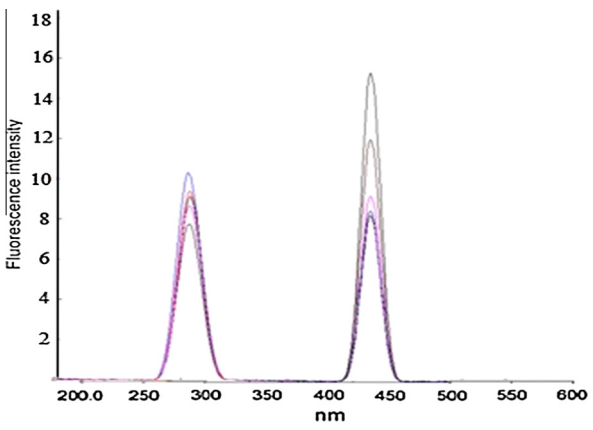

Fig. 2. Spectra of excitation and emission at 284 nm and 446 nm.