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Stability-indicating methods for the determination of piretanide in presence of the alkaline induced degradates

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Abstract

Stability-indicating high performance liquid chromatography (HPLC), thin-layer chromatography (TLC) and first-derivative of ratio spectra (1DD) methods are developed for the determination of piretanide in presence of its alkaline induced degradates. HPLC method depends on separation of piretanide from its degradates on μ-Bondapak C_{18} column using methanol:water:acetic acid (70:30:1, v/v/v) as a mobile phase at flow rate 1.0 ml/min and UV detector at 275 nm. TLC densitometic method is based on the difference in Rf-values between the intact drug and its degradates on thin-layer silica gel. *Iso*-propanol:ammonia 33% (8:2, v/v) was used as a developing mobile phase and the chromatogram was scanned at 275 nm. The derivative of ratio spectra method (1DD) depends on the measurement of the absorbance at 288 nm in the first-derivative of ratio spectra for the determination of the cited drug in the presence of its degradates. Calibration graphs of the three suggested methods are linear in the concentration ranges 0.02–0.3 μg/20 μl, 0.5–10 μg/spot and 5–50 μg/ml, with mean percentage recovery 99.27 ± 0.52, $99,17 \pm 1.01$ and $99.65 \pm 1.01\%$, respectively. The three proposed methods were successfully applied for the determination of piretanide in bulk powder, laboratory-prepared mixtures and pharmaceutical dosage form with good accuracy and precision. The results were statistically analyzed and compared with those obtained by the official method. Validation of the method was determined with favourable specificity, linearity, precision, and accuracy was assessed by applying the standard addition technique.
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Keywords: Stability-indicating; HPLC; TLC; Ratio spectra; Piretanide

1. Introduction

Piretanide, 4-phenoxy-3-(pyrrolidin-1-yl)-5-sulphamoyl benzoic acid, affects renal and cardiovascular function. It is used as a diuretic and antihypertensive drug [1,2]. Piretanide exhibits a native fluorescence so spectrofluorimetric methods were published [3,4]. A few techniques have been applied for its determination in the biological fluids and pharmaceutical preparation including HPLC [5–7], electrochemical [8], and radio-immunoassay [9].

However, TLC and spectrophotometric methods have not been reported previously, although they are simple, direct and economical in cost, time and chemicals. Hence the proposed methods are suitable for routine analysis in quality control laboratories.

The aim of this work was to introduce stability-indicating methods for the determination of piretanide in presence of its degradates without any interference.

2. Experimental

2.1. Apparatus

- HPLC system: waters LC equipped with 600 controller; 486 Tunable absorbance detector, 600 pump, injector valve with constant 20 μl loop; integrator 746 Data Module; column μ-Bondapak C_{18}, 10 μm, 250 × 4.6 mm.
- Densitometer dual wavelength SHIMADZU flying spot CS-9301PC.

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- UV lamp short wavelength 254 nm.
- Thin-layer chromatographic plates precoated with silica gel GF, 10 cm × 10 cm, 0.25 mm thickness, fluorescent at 254 nm (E-Merck, Germany).
- UNICAM UV 300, Thermo spectronic, with vision 32 software, with 1 cm quarts cuvettes, connected to IBM PC computer used for all the absorbance measurements and treatment of data and hp laser Jet 1000 series printer.

2.2. Materials

Piretanide, working standard, was kindly supplied by Hoechst Orient S.A.E. Co., Cairo, Egypt, its purity was found to be 99.29 ± 0.55% according to the European Pharmacopoeia [10].

Arelix tablets (Japan Hexal, 3-1-10 Mita, Minatoku, Tokyo, Japan), each tablet was labeled to contain 6 mg piretanide.

2.3. Reagents

Chromatographic grade, methanol:water:glacial acetic acid (70:30:1, v/v/v) as a mobile phase for HPLC.

Spectroscopic grade, *iso*-propanol:ammonia 33% (8:2, v/v) as a developing mobile phase and methanol as a solvent for TLC and ratio spectra.

2.4. Preparation of the degradates

Fifty milligrams of piretanide was dissolved in 50 ml aqueous sodium hydroxide (pH 9) and transferred into a screw-capped tube. The solution became dark brown with pH 7 after exposure to sunlight for 10 days. Decomposition was assessed by applying TLC using *iso*-propanol:ammonia 33% (8:2, v/v) and by disappearance of the native fluorescence of the intact drug at 335 nm excitation and 415 nm emission in methanol. The solution of the degradates (1 mg/ml) was kept in a refrigerator and used for the preparation of laboratory-prepared mixtures.

2.5. Standard stock solutions

- Piretanide (0.1 mg/ml) in the mobile phase for HPLC or in methanol for ratio spectra.
- Piretanide (1 mg/ml) in methanol for TLC.
- Degradates (0.1 mg/ml) in the mobile phase for HPLC or in methanol for ratio spectra.
- Degradates, (1 mg/ml) in methanol for TLC.

All solutions were stable for at least 1 month if stored in a refrigerator at 4 °C.

2.6. Procedure

2.6.1. Calibration for HPLC method

Aliquots of standard stock solution (0.1 mg/ml) equivalent to 1–15 μg piretanide were transferred into a series of 10 ml volumetric flasks and diluted up to the mark with the mobile phase methanol:water:acetic acid (70:30:1, v/v/v). Twenty microlitres of each solution was injected at flow rate 1.0 ml/min. The effluent was monitored at 275 nm, AUFs equal 0.05 and the chromatograms were recorded. The calibration curve representing the relationship between the recorded area under the peak and the corresponding concentration was plotted and the regression equation was computed.

2.6.2. Calibration for TLC method

Aliquots of standard stock solution (1 mg/ml) equivalent to 0.25–4.5 mg piretanide were transferred into a series of 5 ml volumetric flasks and diluted up to the mark with methanol. Ten microlitres of each solution was applied to TLC plate. The chromatographic chamber was equilibrated with the developing mobile phase *iso*-propanol:ammonia 33% (8:2, v/v) for 10 min prior to use. The plates were developed over a distance of 8 cm and air dried at room temperature. The spots were visualized under UV lamp at 254 nm and the chromatogram was scanned with spectrodensitometer at 275 nm using photo mode: reflection and scan mode: zigzag. The calibration curve representing the relationship between the recorded area under the peak and the corresponding concentration was plotted and the regression equation was computed.

2.6.3. Calibration for 1DD method

Aliquots of standard stock solution (0.1 mg/ml) equivalent to 0.05–0.5 mg piretanide were transferred into a series of 10 ml volumetric flasks and diluted up to the mark with methanol. The zero-order spectrum of each solution was divided by the spectrum of the degradates (45 μg/ml). All spectra were stored in the IBM PC. The first-derivative of each ratio spectra was recorded at range 200–400 nm; band width 1.5 nm; scan speed: intelliscan; data interval: normal; smoothing: high and 1DD values were measured at 288 nm. The calibration curve was plotted and the regression equation was computed.

2.7. Laboratory-prepared mixtures

2.7.1. HPLC method

Aliquots equivalent to 1–15 μg of piretanide solution (0. 1 mg/ml) were transferred into a series of 10 ml volumetric flasks. Ten to ninety percentage of the degradates (0.1 mg/ml) were added to the same flasks and diluted up to the mark with the mobile phase.

2.7.2. TLC method

Aliquots equivalent to 0.25–4.5 mg of piretanide solution (1 mg/ml) were transferred into a series of 5 ml volumetric

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