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Determination of lisinopril using anion exchange chromatography with integrated pulsed amperometric detection

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Abstract

A rapid and practical method for direct detection of lisinopril in anion exchange chromatography (AEC) has been developed with integrated pulsed amperometric detection (IPAD). Dionex AS18 (250 mm × 2 mm) and AG18 (50 mm × 2 mm) columns and 40 mmol/L NaOH solution were used for separation. Multi-step potential waveform parameters were optimized to maximize the signal-to-noise ratio (S/N). Utilizing the optimized waveform, the repeatability (intra-day) precision and intermediate (inter-day) precision were obtained with relative standard deviation (RSD) of 0.74, 0.93, respectively. The limit of quantification (LOQ) and limit of detection (LOD) were found to be 0.37, 0.12 ng/mL, respectively, with the correlation coefficient of 0.9998 over concentration range 0.01–1 μg/mL. The present method was successfully applied to the determination of lisinopril in human plasma. The recoveries of plasma sample spiked by 0.2 μg/mL, 0.8 μg/mL lisinopril were 98.31–103.23% with RSD of 1.41%, 0.61%, respectively.

Keywords: Lisinopril; Integrated pulsed amperometry; Anion exchange chromatography; Angiotensin converting enzyme inhibitor; Plasma

Lisinopril (LN), (2*S*)-1-[(2*S*)-6-amino-2[[(1*S*)-1-carboxy-3-phenylpropyl] amino] hexanoyl]pyrrole-2-carboxylic acid (Fig. 1), an inhibitor of the angiotensin converting enzyme (ACE), is widely used for treating high blood pressure and heart failure [1]. Lisinopril is a subject of monograph in the United States Pharmacopoeia (USP), which recommends an HPLC method for its analysis [2].

A variety of analytical methods have been developed for the determination of lisinopril in pharmaceutical preparations, such as HPLC [3], GC [4], capillary electrophoresis [5,6], spectrophotometry [7–10], spectroflurimertric [8,9], micellar electrokinetic chromatography [10] and polarographic [9]. Methods have been described for determination of lisinopril in biologic fluids including GC [11], HPLC [12–14], LC–MS [13], fluoroninmmunoassay [15,16], radiommuoassay [17], fluorozmatic assay [18] and ion selective electrodes [19].

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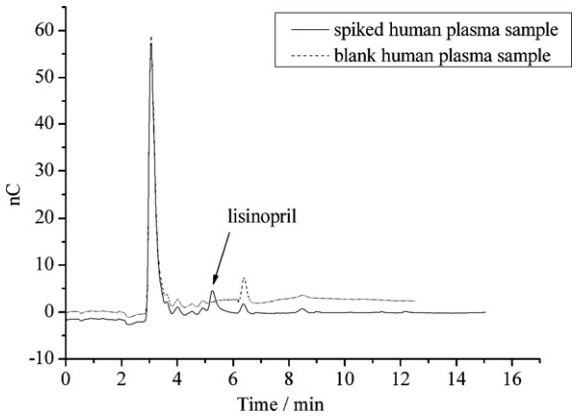

Fig. 1. Typical AEC–IPAD chromatogram of human plasma spiked with 20 μg/mL lisinopril and diluted 1:100 (0.2 μg/mL) with water (—), and blank sample (- - -). Column, Dionex AS18 (250 mm × 2 mm) plus guard column. Eluent, 40 mmol/L NaOH solution at a flow rate 0.25 mL/min.

Lisinopril possesses poor electromagnetic absorbance due to week benzene chromophore; as a consequence, poor sensitivity can be achieved by UV spectrophotometric method. Therefore, most of these assays employ pre-column derivatization reactions for increasing determination sensitivity. However, the preparation of derivatives is a time-consuming procedure, and the derivatization frequently generates products that are unstable. Moreover, peak splitting owing to slow cis–trans isomerization may exist in the RP-HPLC [20]. GC and LC with mass spectrometric detection were widely applied to determination in biological fluids. Being highly sensitive methods, nevertheless still require sophisticated equipments that were not available in many laboratories.

Pulsed electrochemical detection was developed for polar aliphatic compounds, which contain amine, alcohol, or sulfur moieties. Since lisinopril is a polar compound and is present as an anion under alkaline condition, it therefore can be separated by AEC. Further more, it contains primary and secondary amine groups, which are expected to be directly oxidized at gold electrodes under alkaline conditions. In this study, integrated pulsed amperometric detection (IPAD) was applied to detect lisinopril with ion chromatography. It was demonstrated that this method was a rapid and simple one for the determination of lisinopril in human plasma.

1. Experimental

A DX-600 ion chromatography system consisting of a GP50 gradient pump and a LC 25 chromatography oven was connected with an ED50A electrochemical detector was used in this study. Dionex AS18 (250 mm × 2 mm) and AG18 (50 mm × 2 mm) columns packed with anion-exchange resin were used as the separation columns. The concentration of NaOH solution used as eluent was 40 mmol/L.The analysis was performed at 30 °C with the flow rate set at 0.25 mL/min. In all analyses, 10 μL was injected. The detection was carried out by a pulsed amperometry cell equipped with a working gold electrode and a combined pH Ag/AgCl reference electrode.

Aliquots of 400 μL plasma were diluted with 400 μL water in a 4 mL polypropylene tube. 800 μL acetonitrile were then added. The mixtures were vortex mixed for about 60 s then centrifuged at 1000 r/min for 10 min.The clear supernatant was pipetted and was evaporated under vacuumina stream of nitrogen at about 30 °C to almost dryness and the volume was completed to 400 μL with water. The sample was then loaded to the solid phase extraction (SPE) cartridge. The cartridge was preconditioned by washing with 3 mL water, 3 mL acetonitrile then another 2 mL water. After loading the sample, the cartridge was washed with 2 mL water then dried by using airflow under vacuum. The sample was filtered through a 0.45 μm syringe filter then diluted 1:100 with water, 10 μL were directly injected to the system.

2. Results and discussion

Waveform optimization was performed by injecting a standard solution of lisinopril and plotting the amperomertric single-to-noise ratio as a function of the parameter to be optimized. The waveform consists of three distinct regions