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Determination of josamycin in rat plasma by capillary electrophoresis coupled with post-column electrochemiluminescence detection

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Abstract

A novel determination method for josamycin (JOS) based on capillary electrophoresis–electrochemiluminescence detection has been described. In this study, platinum disk electrode (300 μm in diameter) was used as a working electrode and the conditions affecting separation and detection were investigated in detail. Under optimal condition: 40 cm separation capillary (75 μm i.d.); 1.25 V applied potential on the Pt disc of the ECL detector cell; 5 mM $Ru(bpy)_3^{2+}$ and 50 mM phosphate buffer (pH 7.5) in the detection cell; 12 kV separation voltage; 8 s injection time; 10 kV injection voltage and 15 mM running buffer (pH 7.5), calibration curve was linear over the range from 10 ng/mL to 5.0 μg/mL with a detection limit of 3.1 ng/mL at a signal-to-noise ratio of 3. The method can be successfully applied for the determination of josamycin in rat plasma in 6 min and the extraction recoveries with spiked plasma samples were over 92%.

Keywords: Capillary electrophoresis; Electrochemiluminescence; Josamycin; Tris(2,2′-bipyridyl) ruthenium(II); Plasma

1. Introduction

Josamycin (JOS) is a macrolide antibiotic (see Fig. 1), effective against mycoplasma, gram positive cocci and bacilli, and certain gram negative organisms [1]. Like other macrolide antibiotics, JOS is a lipophilic molecule with a central lactone ring of 16 atoms to which several amino and sugar moieties are bound [2,3].

Most of the methods reported for the analysis of JOS in biological fluids, tissues, eggs and milk rely on the use of liquid chromatography [1,4–9] in addition to capillary electrophoresis [10]. A voltametric method based on the cathodic reduction of the diene group has been proposed for JOS [11]. In addition, two reports are available yet on the kinetic spectrophotometric determination of JOS in dosage forms and spiked biological fluids [12,13]. LC–MS is a very sensitive technique for macrolide antibiotics [1], but in these cases, the analytical time is long and the consumption of sample and reagents is not minimum, interferences from other compounds present in complex biological samples, and extensive instrument is also required. So there still exists a need for improved detection techniques of JOS and related macrolide antibiotics. Lalloo et al. mainly considered optimal separation conditions of JOS using CE [10]. But in this paper, not only to optimize the conditions of separation, but also to optimize the detection conditions of ECL and determine JOS in rat plasma.

Capillary electrophoresis (CE) technique has been developed for over 20 years. CE is an important and powerful analytical tool due to its high efficiency, relatively short analysis time, and small sample volume [14]. Electrochemiluminescene (ECL) detection with $Ru(bpy)_3^{2+}$ is a viable alternative for CE detection. ECL has been applied to a variety of fields such as biosensors, immunoassays, and flow injection analysis [15]. CE with ECL detection using $Ru(bpy)_3^{2+}$ has been studied since the mid-to-late 1990s for the determination of a variety of analytes which were listed in some reviews [16–30]. Compared with the previous report, the combination of CE with ECL showed several advantages: easy operation, high selectivity to the specific analyte, fast analysis speed, low-reagent consumption, etc. [31].

In this work, the CE–ECL is mainly concerned with the determination of JOS containing tertiary amine group recently

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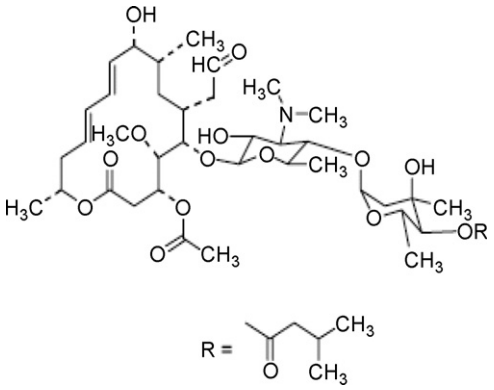

Fig. 1. Molecular structure of josamycin.

because the tertiary amine has high-enhancement effect on the $Ru(bpy)_3^{2+}$ ECL. Based on this observation, CE with ECL detection is applied to the determination of JOS in rat plasma.

2. Experimental

2.1. Reagents and chemicals

All reagents used were of analytical grade. Double-distilled water (DDW) was used throughout. JOS was obtained from the National Institute for The Control of Pharmaceutical and Biological Products (Beijing, China). Tris(2,2′-bipyridyl) ruthenium(II) chloride hexahydrate was purchased from Alfa Aesar (A Johnson Matthey Company, Ward Hill, MA, USA) and used without further purification. All solutions were prepared with DDW and stored in a refrigerator at 277 K. Working standard solutions were freshly prepared by precise dilution of stock solutions with DDW. The buffer used throughout the study was sodium phosphate system (Na_2HPO_4/NaH_2PO_4, Hunan Reagent Company, Hunan, China). Prior to CE analysis, the required sample solutions and phosphate buffer were filtered through 0.45 μm membrane filters (Shanghai Xinya Purification Material Factory, Shanghai, China).

2.2. Apparatus and equipments

The CE–ECL system (a high-voltage power supply, an electrical potentiostat, a multifunction chemiluminescence detector, and a multichannel data collection analyzer) was produced by Xi'an Remex Electronic Science-Tech Co. Ltd. (Xi'an, China). The output ECL intensity was amplified and recorded in a Pentium 4 PC using the MPI-B software.

The CE–ECL detection cell has been described previously [19]. The end-column ECL cell was composed of a three-electrode system, with 300 μm-diameter platinum disk as the working electrode, a Pt wire as the counter electrode, and a Ag/AgCl electrode as the reference electrode. The surface of the working electrode was polished with 0.3 μm-alumina powder and cleaned with water in an ultrasonic cleaner before use. A reactivation process to eliminate the oxide layer on the Pt electrode was performed by scanning the applied potential on the Pt disk from −0.5 to 0.0 V (vs. Ag/AgCl) for 10 cycles. The performance of the working electrode was stable for at least 2 months after the electrochemical treatment. All electrophoretic experiments were performed with a 40 cm uncoated silica capillary (75 μm i.d. and 375 μm o.d.) (Yongnian Optical Fiber Co., Hebei, China).

PHSJ-4A pH meter (Shanghai Precision Science Instrument Co., Ltd., Shanghai, China), SK3200H Ultrasonic Cleaner (Shanghai Kudos Ultrasonics Instrument Co., Ltd., Shanghai, China). TGL-16G-A Centrifuge (Shanghai Anting Science Instrument Factory, Shanghai, China). BF-2000M Nitrogen Blow Instrument (Beijing Bafang Century Technology Co., Ltd., Beijing, China).

2.3. Sample preparation and extraction procedure

The blank plasma samples, for method development and validation were obtained from rats weighing 300 g which were purchased from Guilin Medical College, heparinized and centrifuged for 10 min at 3500 r/min to separate the plasma. Plasma samples were stored at 253 K until assay.

Preparation for rat plasma sample was well documented by previous study in our laboratory [32]. Female rats were anesthetized with using ethyl ether, and polyethylene tubes were inserted into the femoral vein and artery. After recovery from anesthesia, JOS (20 mg/kg) dissolved into a physiological salt solution (NaCl: 135 mM, $CaCl_2$: 1.8 mM, $MgCl_2$: 1.0 mM, KCl: 5.4 mM, $NaHCO_3$: 11.9 mM) was intravenously injected. Blood samples were collected from femoral artery at 5 min after administration and immediately centrifuged to obtain plasma. The plasma samples were then extracted as described above.

Plasma samples (200 μL) were pipetted into clean centrifugation tubes, after addition of 2 mL of ethyl acetate, the mixture was made alkaline with 5 μL of 1 M NaOH, and then shaken for 5 min, after centrifugation (10 min, 3000 r/min), the upper layer was transferred into a tube and evaporated to dryness under a stream of dry nitrogen at 353 K. Next, the inner wall of the tube was rinsed with 200 μL of methanol to concentrate the sample and evaporated off. The dry residue was dissolved in 200 μL water and measured.

2.4. Electrophoresis procedure

Before each run, the capillary was flushed with water and running buffer for 10 min, respectively. The detection reservoir was filled with 300 μL of 5 mM $Ru(bpy)_3^{2+}$ and 50 mM pH 7.5 phosphate buffer solution before analysis and replaced every 3 h to eliminate depletion effect. Injections were performed by electromigration at a constant voltage of 10 kV for 8 s (0.2 μL). During the experiment, a 15 kV separation voltage was applied across the capillary and the potential of the photomultiplier tube (PMT) was set at 800 V.

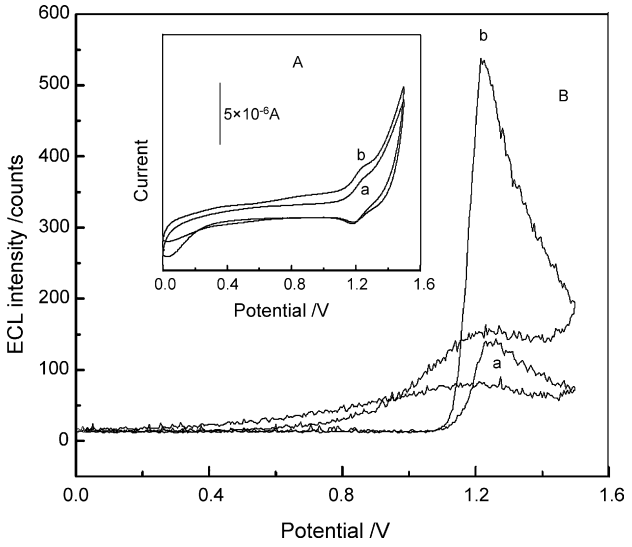

Fig. 2. Cyclic voltammograms (A) and the corresponding ECL intensities (B). Sample: 2.5 mM $Ru(bpy)_3^{2+}$ without (a) and with (b) 1.0×10^{-6} g/mL JOS. Conditions: 50 mM buffer with pH 7.8 and 2.5 mM $Ru(bpy)_3^{2+}$ in the ECL cell. Scan rate: 100 mV/S.

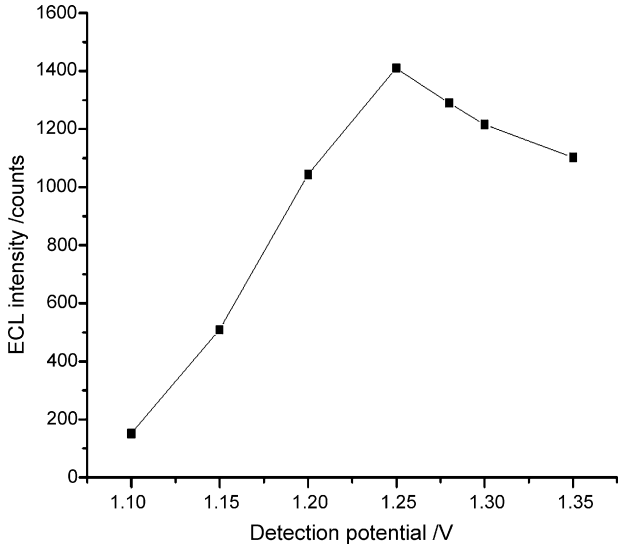

Fig. 3. The effect of detection potential on ECL intensity. Conditions: sample, 1.0×10^{-6} g/mL JOS; detection potential, 1.25 V; electrokinetic injection, 10 kV × 6 s; separation buffer, 10 mM pH 7.8 phosphate buffer; separation voltage, 15 kV; 5 mM $Ru(bpy)_3^{2+}$ and 50 mM pH 7.8 phosphate buffer in ECL cell.

3. Results and discussion

3.1. Cyclic voltammetry of $Ru(bpy)_3^{2+}$ and josamycin

The electrochemiluminescene (ECL) intensity is dependent on the rate of the light-emitting chemical reaction which is in turn dependent on the potential applied to the electrode. Cyclic voltammograms (CV; Fig. 2(A)) and the corresponding ECL intensity (Fig. 2(B)) were recorded.

Under the CV conditions, the rise of ECL intensity of JOS solution and buffer solution (Fig. 2(B)) started at about 1.10 V and increased significantly with the increase of potential. The ECL intensity of JOS was three times higher than the background noise caused by the direct oxidation of $Ru(bpy)_3^{2+}$ at the high-applied potential of 1.25 V. These observations indicated that JOS can react with the ruthenium species in the electrochemiluminescence process and it can enhance the emitted light intensity.

After CE separation, the JOS contained in CE effluent are in contact with $Ru(bpy)_3^{2+}$ at the electrode surface and a electropherogram is obtained because the type and concentration of JOS in the CE effluent is not constant. The ECL intensity reaches its maximum in the potential region of the direct oxidation of $Ru(bpy)_3^{2+}$. The generation of $Ru(bpy)_3^{2+*}$ related ECL is formed through the redox reaction of JOS intermediate (radical) [16]. Because the JOS containing tertiary amine have similar electrochemical and ECL behavior to tripropylamine (TPA), the ECL of JOS route and mechanism in CE–ECL procedure can be learned from $Ru(bpy)_3^{2+}$/TPA system as given below [31,33]:

$$Ru(bpy)_3^{2+} - e^- \rightarrow Ru(bpy)_3^{3+} \quad (1)$$

$$JOS - e^- \rightarrow JOS^{\bullet +} \rightarrow JOS^{\bullet} + H^+ \quad (2)$$

$$Ru(bpy)_3^{3+} + JOS^{\bullet} \rightarrow Ru(bpy)_3^{2+*} + \text{product} \quad (3)$$

$$Ru(bpy)_3^{2+*} \rightarrow Ru(bpy)_3^{2+} + h\nu \quad (4)$$

ECL is produced from the concomitant oxidation of $Ru(bpy)_3^{2+}$ and JOS. The JOS radical cation loses a proton from α-carbon to form the strongly reducing intermediate, $JOS^{\bullet}$ (Eq. (2)). And then the radical reduces $Ru(bpy)_3^{3+}$ to $Ru(bpy)_3^{2+*}$, which emits light. As the tertiary amine derivatives, the electron-withdrawing/donating character of the R group attached to N atom in analytes influences the ECL efficiency of $Ru(bpy)_3^{2+}$/analytes [34].

3.2. Optimization of detection conditions

The relationship between ECL intensity and the applied potential was investigated in this work. It was found that with the increase of the applied potential, the ECL intensity increased and reached a maximum value at 1.25 V (Fig. 3), then decreased slightly. Therefore, in the following experiment, the applied potential was set at 1.25 V.

The optimal concentration of $Ru(bpy)_3^{2+}$ in the detection cell is one of the most important detection parameters. A low concentration of $Ru(bpy)_3^{2+}$ leads to a low-background noise. But the detection sensitivity increases with the concentration of $Ru(bpy)_3^{2+}$. The concentration of $Ru(bpy)_3^{2+}$ in the detection cell was well studied in the literature [35]. Although, higher concentration of $Ru(bpy)_3^{2+}$ showed larger response, it also yielded more noise. Another investigation of the concentration of the buffer in the detection cell was also performed. In our work, 5 mM $Ru(bpy)_3^{2+}$ and 50 mM sodium phosphate produced a higher S/N value and was used in all the experiments shown [36].

Table 1
Effect of the phosphate buffer pH on ECL intensity

pH	Intensity[a]/counts	Intensity[b]/counts
6.0	352	1044
6.5	678	1403
7.0	1090	1524
7.5	1432	1660
8.0	1314	1483
8.5	1043	1114
9.0	904	964

[a] For effect of the phosphate buffer pH (in ECL cell) on ECL intensity. Operating conditions: sample, 1.0×10^{-6} g/mL JOS; detection potential, 1.25 V; electrokinetic injection, 10 kV × 6 s; separation buffer, 10 mM (pH 7.8) phosphate buffer; separation voltage, 15 kV; 5 mM $Ru(bpy)_3^{2+}$ and 50 mM phosphate buffer in ECL cell.

[b] For effect of the separation buffer pH on ECL intensity. Operating conditions: sample, 1.0×10^{-6} g/mL JOS; detection potential, 1.25 V; electrokinetic injection, 10 kV × 6 s; separation buffer, 10 mM phosphate buffer; separation voltage, 15 kV; 5 mM $Ru(bpy)_3^{2+}$ and 50 mM (pH 7.5) phosphate buffer in ECL cell.

3.3. Optimization of buffer pH

The pH of detection buffer has a significant effect on the ECL response. The previous work indicated that $Ru(bpy)_3^{2+}$ has a good ECL efficiency in a weak basic solution because of the deprotonation of the amine species to form a reducing free radical intermediate and the reaction between radical intermediate and ruthenium species [31,37–39]. Also, the pH of separation buffer influences electroosmotic flow (EOF) and the analyte ionization and thus, the separation efficiency. Moreover, if the pH value of separation buffer is different from that of detection buffer, a pH gradient will be noticed along the capillary; so, to eliminate the effect of the difference between detection buffer and separation buffer, two buffers with same pH were used [26].

Table 1 gave the effects of detection buffer pH and separation buffer pH on the detection intensity, respectively. Similar to the amine ECL reaction, both the highest ECL intensity of JOS for detection buffer and separation buffer was at pH 7.5. To maintain good sensitivity and a suitable analysis time, both the detection buffer and the separation buffer with pH 7.5 were used.

3.4. Optimization of separation conditions

Many factors primarily the double layer thickness and ionic strength, hence the separation voltage and migration velocity, etc. were influenced by the buffer concentration in CE. The ECL intensity in the present work also changed as a function of the buffer concentration. With the increase of buffer concentration, ECL intensity ascends first and then descends with a turning point of 15 mM (Fig. 4). In the following experiments, 15 mM (pH 7.5) phosphate buffer was used as the CE running buffer.

The influence of separation voltage on the ECL intensity was carried out from 8 to 18 kV (Fig. 5). High voltages reduced the analysis time and sharpened the peaks by minimizing zone broadening. Theoretically, resolution and efficiency are directly proportional to the applied voltage [39]. However, the inability of the system to remove the excess Joule heat generated at high

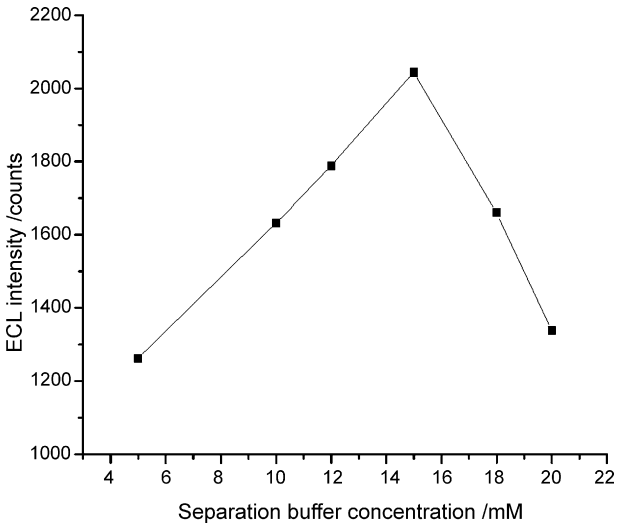

Fig. 4. The effect of concentration of the separation buffer on ECL intensity. Detection potential: 1.25 V; separation buffer: pH 7.5 phosphate buffer; other conditions as in Fig. 3.

voltages, results in peak broadening and a decrease in efficiency and resolution [40]. ECL intensity increased with separation voltage increased up to 12 kV, then it dropped as the voltage was further increased. We chose 12 kV as a separation voltage in this experiment to ensure high-ECL intensity as well as good resolution.

3.5. Effect of injection time and voltage

Fig. 6 shows the effect of injection voltage on ECL intensity and theoretical plate number (N) as injection time set at 6 s, and Fig. 7 shows that ECL intensity and N are influenced by the injection time ranging from 2 to 14 s. Theoretical plate efficiencies of JOS are calculated using the equation: $N = 5.54(t_R/W_h)^2$, where t_R is the migration time and W_h is the width at half the

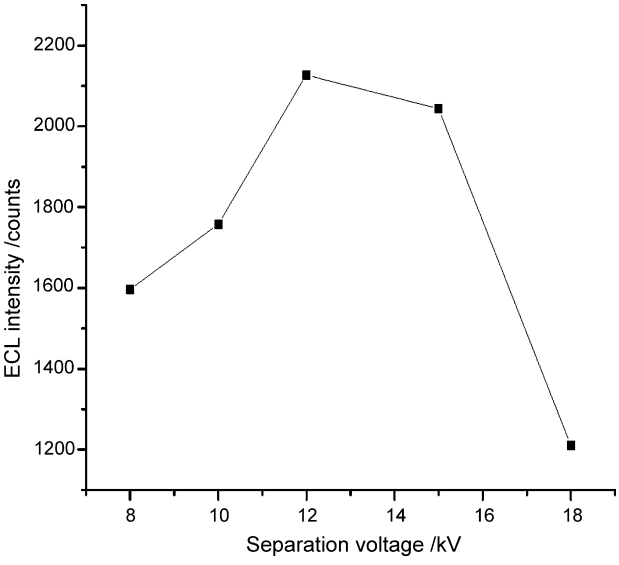

Fig. 5. The effect of separation voltage on ECL intensity. Separation buffer: 15 mM pH 7.5 phosphate buffer; other conditions as in Fig. 4.

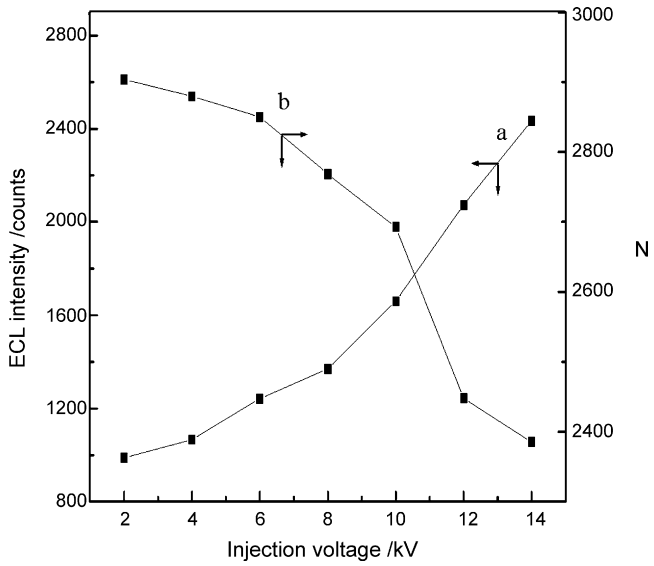

Fig. 6. Dependence of the ECL intensity (a) and separation efficiency (b) on the injection voltage. Separation voltage for 12 kV; other conditions as in Fig. 5.

maximum peak height. As illustrated in Figs. 6 and 7, long injection time and high voltage led to strong ECL signal and low-separation efficiency due to the introduction of more analyte in the detection cell. However, when shorter injection time and lower injection voltage were used, it was difficult to obtain favorable ECL intensity though high-column efficiency could be achieved. So, electrokinetic injection for 8 s at 10 kV was used as a compromise [37].

3.6. Linearity, detection limit, and reproducibility

The optimized experimental conditions were 40 cm separation capillary (75 μm i.d.), 1.25 V applied potential on the Pt disc of the ECL detector cell, 5 mM $Ru(bpy)_3^{2+}$ and 50 mM phosphate buffer (pH 7.5) in the detection cell, 12 kV separation voltage, 8 s injection time, 10 kV injection voltage, and 15 mM running buffer (pH 7.5). Calibration curve for JOS is linear over the concentration range from 10 ng/mL to 5 μg/mL with a regression curve of $y = 23382x + 1001$ ($r^2 = 0.9985$, y for peak area, x concentration unit for μg/mL). The detection limit was 3.1 ng/mL with a signal-to-noise ratio of 3. The relative standard deviations (R.S.D.) of the ECL intensity and the migration time for eleven consecutive injections of 1.0 μg/mL JOS were 3 and 2.2%, respectively.

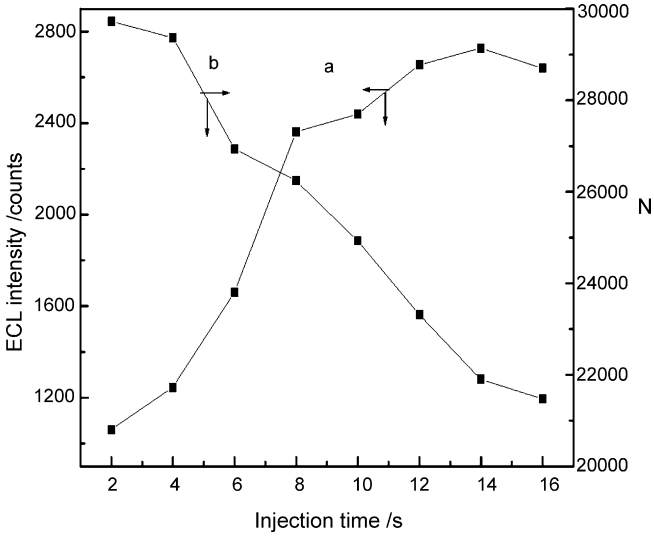

Fig. 7. Effect of the ECL intensity (a) and separation efficiency (b) on the injection time. Injection voltage for 10 kV; other conditions as in Fig. 6.

Table 2
Analytical results of JOS in rat plasma ($n = 5$)

Sample no.	Plasma content (μg/mL)	Added (μg/mL)	Found (μg/mL)	Recovery (%)	R.S.D. (%)
1	3.81	0.500	4.28	94.0	4.1
2	3.86	0.500	4.32	92.0	2.8
3	3.85	0.500	4.34	98.0	3.9

3.7. Detection of josamycin in rat plasma

The proposed CE–ECL method was employed for the determination of JOS in rat plasma samples. Because electrokinetic injection mode was employed in this work, the ionic strength of sample matrix would influence the injection of samples. On the other hand, some organic compounds in plasma might influence the ECL reaction. Therefore, an extraction procedure was performed to remove the ions and some organic compounds in plasma to obtain a clear electrophoretic sample profile, high-detection sensitivity and good reproducibility [27]. The extraction procedure was shown in the experimental and the electropherograms were shown in Fig. 8. Table 2 shows the analytical results of JOS in rat plasma. To determine the recovery of the method, independent plasma samples of rat were spiked at 0.500 μg/mL. All samples were submitted to the analytical procedure described above. The recoveries of JOS in plasma samples at different concentrations were found to be in the range of 92–98%. The R.S.D. of ECL peak area was less than 5%. JOS

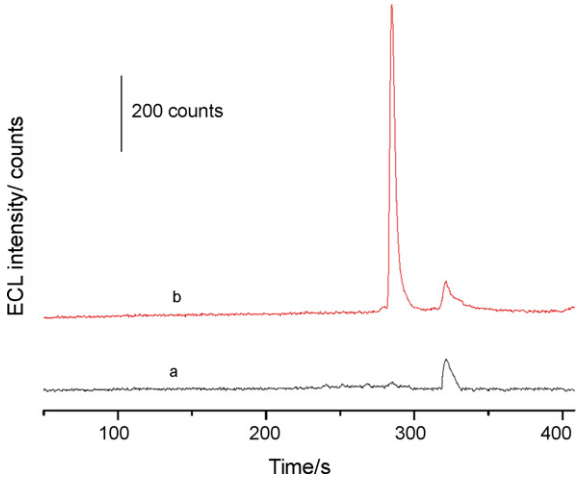

Fig. 8. CE–ECL electropherograms of (a) blank plasma sample and (b) the plasma sample spiked with 0.250 μg/mL JOS under optimal conditions.

concentration in plasma at 5 min after intravenous administration of JOS to rat (20 mg/kg) was an average of 3.84 μg/mL.

4. Conclusions

The article described a simple, rapid, economical and sensitive CE–ECL method for the determination of JOS in rat plasma. Detection limit of 3.1 ng/mL was achieved. Average recovery above 92% with R.S.D. below 5% was obtained for JOS in rat plasma. Therefore, the method may be suitable for the investigation of the pharmacokinetic characteristics of JOS in small animals such as rats. This CE–ECL system can be applied to other compounds detectable by $Ru(bpy)_3^{2+}$ in both biological and non-biological samples.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jchromb.2007.09.014.

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