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Optimization of Electrochemiluminescence Experimental Conditions for Metoclopramide Determination Based on Response Surface Methodology

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Based on the strong enhancement effect of metoclopramide on the electrochemiluminescence signal of $Ru(bpy)_3^{2+}$ on the platinum electrode, a novel method for the sensitive detection of metoclopramide was established. Based on the single factor experiments, the interactions among the three factors were investigated by response surface methodology. Finally, the optimal experimental conditions for the determination of metoclopramide in a limited number of experiments are: detection potential 1.18 V, phosphate buffer solution concentration 39.82 mmol/L and pH 7.69. These conditions can improve the sensitivity of the method by 4.5%, which is very important for the determination of trace components. The detection limit (3σ) of the method was 4.0×10^{-3} mg/L, the linear range was 0.02—36.56 mg/L, the correlation coefficient was 0.9991, and the recoveries were 97.3%—102.4%. This method is simple, rapid, sensitive and with less injection.

Keywords: Electrochemiluminescence; Metoclopramide; Response surface methodology; Optimization

1. INTRODUCTION

Metoclopramide (MCL) is a water-soluble derivative of p-amino benzoic acid. It can block dopamine receptor and act on the chemical induction area of delayed brain vomiting, and play a role in central antiemetic effect. At the same time, it can strengthen the movement of stomach and upper intestinal segment, promote small intestine peristalsis and emptying, relax pyloric sinus and duodenal crown, improve food passing rate, and play a role in peripheral antiemetic effect [1-7]. MCL was synthesized and used in clinic in 1961. It is the first gastrointestinal motility drug. In addition, there are many clinical applications to treat headache [8-18], as well as examples to treat angina [19] and promote lactation [20]. In recent years, with the wide application of MCL, many side effects have been

reported [21-29]. For example, MCL may block the dopamine receptor and make it relatively hyperactive, leading to extra pyramidal reactions, which are manifested as muscle tremor, head backward leaning, torticollis, paroxysmal upward gaze of both eyes, dysphonia, ataxia and other symptoms. Another example is that the United States Food and Drug Administration (FDA) believed that the long-term use of MCL has been proved to be related to tardive dyskinesia, which is manifested in the body's involuntary and repetitive exercise. For this reason, FDA requires all manufacturers of MCL to indicate the risk black frame warning of long-term or high-dose application on their drug labels. It can be seen that the development of a rapid, accurate, sensitive and simple MCL analysis method is of great significance for monitoring the risk of its use.

The main methods for the determination of MCL are liquid chromatography – ultra violet (LC-UV) [30-36], liquid chromatography -mass spectrometry (LC-MS) [37-39], spectrophotometry [40-42], electrochemistry [43-45], fluorometry [46] and capillary electrophoresis (CE) [47]. Electrochemiluminescence (ECL) based on Ru(bpy)32+ is a highly sensitive and selective method for the determination of amines [48-57], especially those compounds containing tertiary amino group. There is a primary amino group, a secondary amino group and a tertiary amino group in the molecular structure of MCL, so it is feasible to determine them by ECL. Response surface methodology (RSM) is a statistical method to solve multivariable problems by using appropriate experimental data to find the optimal process parameters [58,59]. There are many reports in the literature about the optimization of experimental conditions by RSM [60-65].

In our previous experiments, it was found that the weak ECL signal of $Ru(bpy)_3^{2+}$ on the platinum electrode could be strongly sensitized by MCL under the appropriate electrolytic potential. Based on this, a sensitive method for the determination of MCL could be established. However, the change of parameters of experimental conditions had a great influence on the luminescence signal. In this paper, on the basis of single factor experiments, RSM is used to investigate the interactions of different factors. It is expected to improve the sensitivity of the method by optimizing the experimental conditions of ECL analysis.

2. EXPERIMENTAL

2.1. Materials and Reagents

Tris (2,2'-bipyridyl) ruthenium (II) dichloride hexahydrate ($Ru(bpy)_3Cl_2 \cdot 6H_2O$) was purchased from Alfa Aesar (Johnson Matthey, USA). Disodium hydrogen phosphate (Na_2HPO_4) and sodium dihydrogen phosphate (NaH_2PO_4) were all of analytical reagent gradeand were purchased from Beijing Chemical Factory (Beijing, China). Metoclopramide standard substance was purchased from National Institutes for Food and Drug Control (Beijing, China).

2.2. Solutions preparation

$Ru(bpy)_3^{2+}$ solutions were prepared with $Ru(bpy)_3Cl_2 \cdot 6H_2O$ and secondary distilled water.

Phosphate buffer solution (PBS) has good stability, easy preparation and wide pH range. Therefore, phosphate buffer solution is chosen as the experimental environment. PBS was prepared with disodium hydrogen phosphate, sodium dihydrogen phosphate and secondary distilled water. Standard solutions of metoclopramide were prepared with its standard substance and secondary distilled water. All solutions used in the experiment must be filtered through a 0.22 μm cellulose acetate membrane.

2.3. Apparatus

ECL was performed on a MPI-B multi-parameter chemiluminescence analysis test system (Xi'an Remex analytical instruments Co., Ltd., Xi'an, China). Cyclic voltammetry and potentiostatic method were carried out in a three electrodes system with a platinum working electrode of 500 μm in diameter, an Ag/AgCl reference electrode of 300 μm in diameter and a platinum wire auxiliary electrode of 1 mm in diameter. The solution to be tested was injected into the test cell through a microlitre syringe, and the $Ru(bpy)_3^{2+}$- PBS in the test cell was renewed every 3 hours.

3. RESULTS AND DISCUSSION

3.1. Single factor experiment of detection conditions

3.1.1 Detection potential

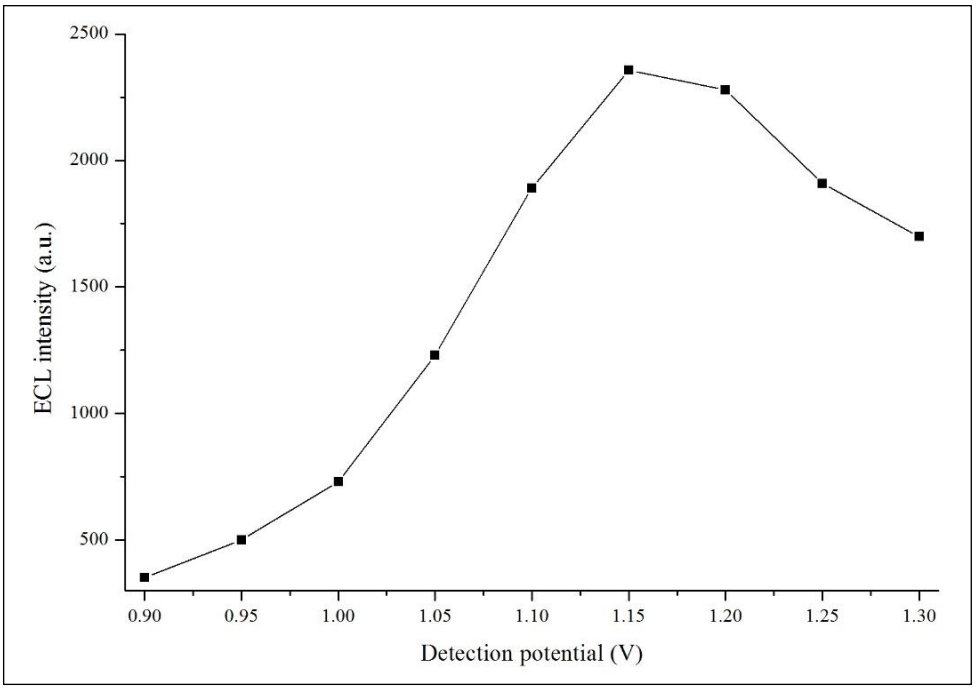

Figure 1. Effect of detection potential on ECL intensity under 5.0 mmol/L $Ru(bpy)_3^{2+}$ and 40 mmol/L PBS (pH = 7.5).

The detection potential of the working electrode has a great influence on ECL intensity.

Therefore, it is necessary to study the detection potential in order to obtain high sensitivity. Figure 1 shows the effect of detection potential in the range of 0.90–1.30 V (vs. Ag/AgCl) on ECL intensities of 2.5 mg/L MCL. As you can see, their ECL intensities were weak when the detection potential is lower than 1.0 V, because $Ru(bpy)_3^{2+}$ can't be oxidized at that potential [48]. The ECL intensities firstly increased and then decreased with the detection potential from 0.90 to 1.30 V (vs. Ag/AgCl). The ECL signals reached maximum at 1.15 V.

3.1.2 pH of PBS in ECL cell

Because the ECL reaction between $Ru(bpy)_3^{2+}$ and alkylamine is greatly affected by pH of PBS, the influence of pH on ECL intensity can be evaluated by changing the pH of PBS. Figure 2 shows the effect of pH of PBS on ECL intensities of 2.5 mg/L MCL. The ECL intensities increased with pH from 5.5 to 7.5 and then decreased when pH was higher than 7.5. The possible reason is that when the pH value is small, partial protonation of metoclopramide resulted in less ECL intensity. As the pH value increases, the degree of protonation decreases, and the ECL intensity increases continuously [49]. Maximum ECL intensities appeared at pH 7.5. Therefore, 7.5 can be used as the initial pH value of buffer.

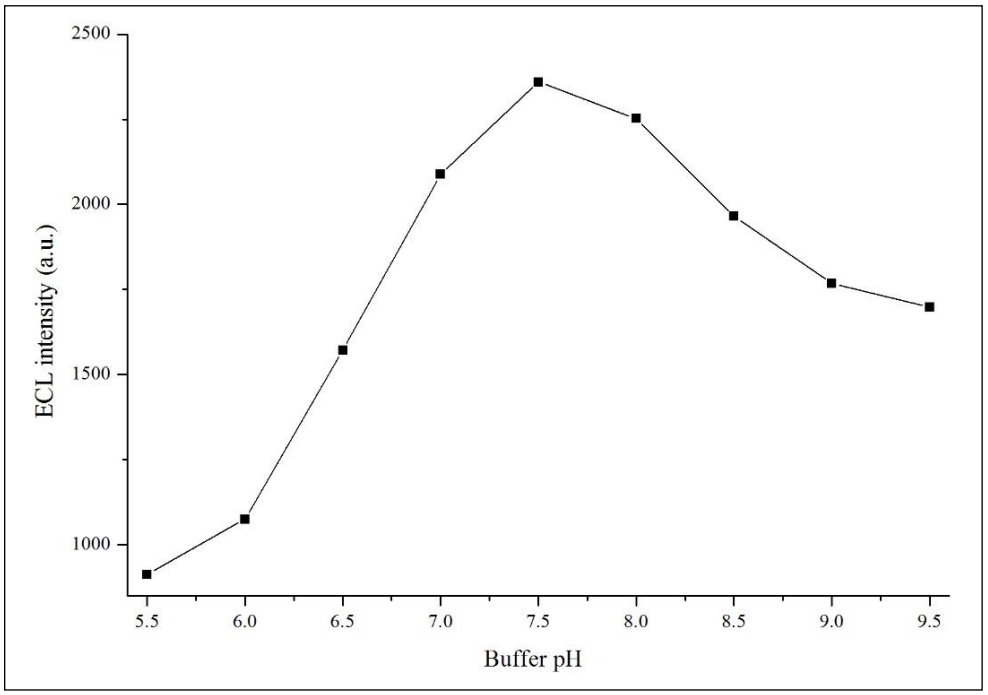

Figure 2. Effect of pH of PBS on ECL intensity under 1.15 V detection potential, 5.0 mmol/L $Ru(bpy)_3^{2+}$ and 40 mmol/L PBS.

3.1.3 Concentration of PBS in ECL cell

The concentration of PBS in the detection cell is also found to affect the ECL intensity. This may be because the influence of ionic strength on the oxidation of MCL by $Ru(bpy)_3^{3+}$ [50]. Figure 3

shows the effect of concentration of PBS in the range of 20–60 mmol/L on ECL intensities of 2.5 mg/L MCL. The maximum ECL intensity appeared when the concentration of buffer was 40 mmol/L.

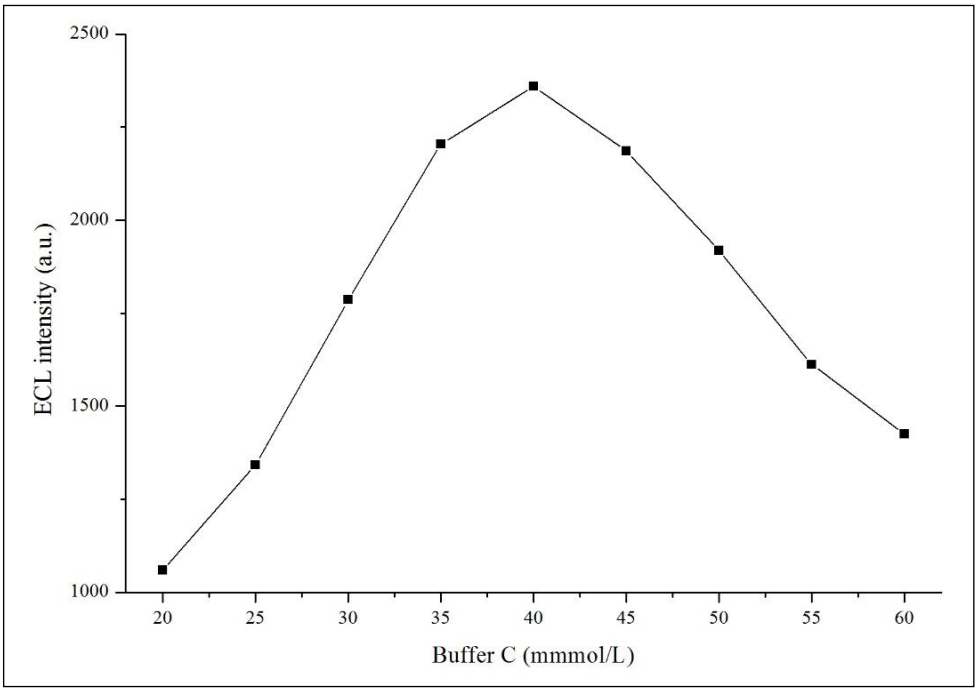

Figure 3. Effect of concentration of PBS on ECL intensity under 1.15 V detection potential, 5.0 mmol/L $Ru(bpy)_3^{2+}$ and pH of PBS at 7.5.

3.2. Optimization of detection conditions by RSM

3.2.1 Box-Behnken test

The detection potential of the working electrode, pH of PBS and concentration of PBS were used as research factors. The ECL intensity was used as the response value. According to the results of single factor experiment, three factors and three levels (see table 1) were used to carry out the Box-Behnken test design.

Table 1. Factors and levels of the Box-Behnken test design.

Levels\Factors	Detection potential (V)	pH of PBS	Concentration of PBS (mmoL/L)
-1	1.10	7.0	35
0	1.15	7.5	40
1	1.20	8.0	45

The results of 17 response surface design trials (12 edge points plus 5 center points in Box-Behnken test design)for 2.5 mg/L MCL are shown in Table 2.

Table 2. Response surface design and experimental results.

Number	Detection potential (V)	pH of PBS	Concentration of PBS (mmoL/L)	ECL intensity (a.u.)
1	1.15	8.0	35	2187
2	1.15	7.0	35	1868
3	1.15	8.0	45	2208
4	1.10	7.5	35	1610
5	1.15	7.0	45	1865
6	1.20	7.5	45	2201
7	1.15	7.5	40	2360
8	1.15	7.5	40	2358
9	1.20	7.5	35	2257
10	1.10	7.0	40	1525
11	1.20	7.0	40	2058
12	1.15	7.5	40	2359
13	1.15	7.5	40	2358
14	1.10	7.5	45	1635
15	1.10	8.0	40	1655
16	1.15	7.5	40	2360
17	1.20	8.0	40	2289

3.2.2 Interaction among factors

The 3D surfaces and contours are plotted by Design Expert software, as shown in figure 4-figure 6. Each figure represents the influence of the interaction of two independent variables on ECL intensity.

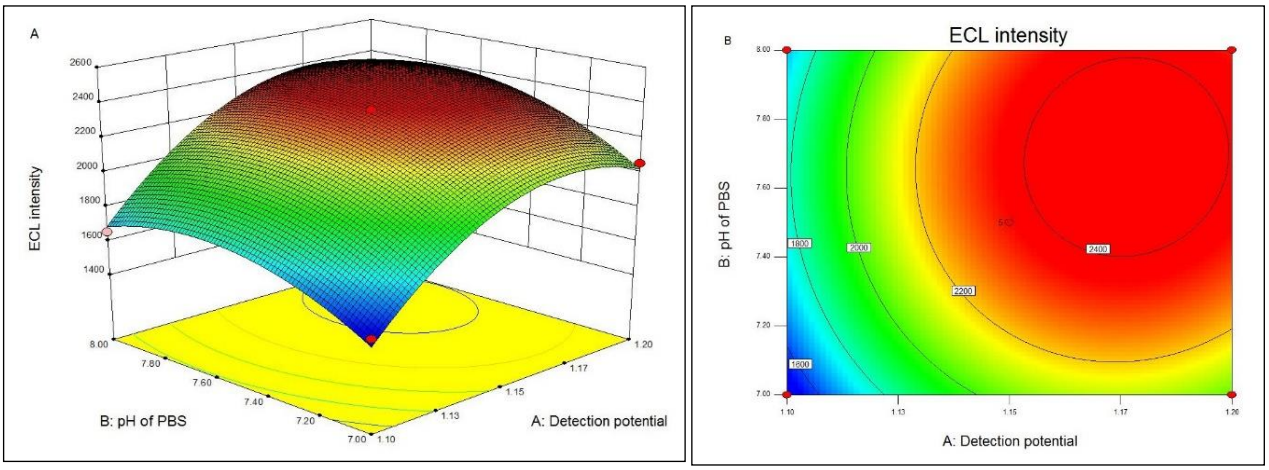

Figure 4. Effect of interaction of detection potential and pH of PBS on ECL intensity.

Figure 4 is 3D surface (A) and contour (B) of the effects of the interaction of detection potential and pH of PBS on the ECL intensity. With the increase of detection potential and pH of PBS,

the ECL intensity increases. When the detection potential reaches 1.18 V and the pH of PBS reaches 7.69, the ECL intensity reaches its maximum. When the detection potential and pH of PBS continue to increase, the ECL intensity begins to decrease.

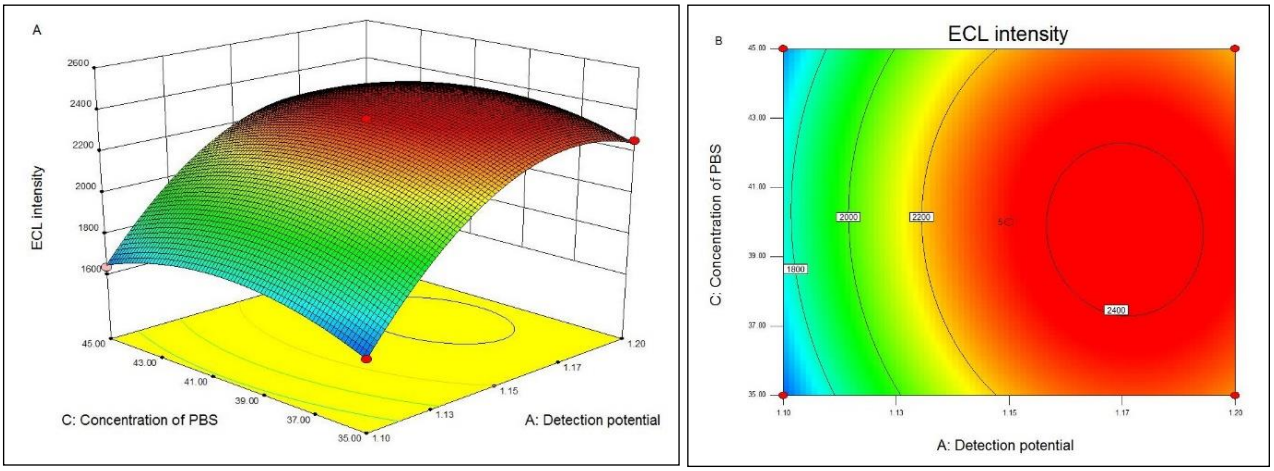

Figure 5. Effect of interaction of detection potential and concentration of PBS on ECL intensity.

Figure 5 is 3D surface (A) and contour (B) of the effects of the interaction of detection potential and concentration of PBS on the ECL intensity. With the increase of detection potential and concentration of PBS, the ECL intensity increases. When the detection potential reaches 1.18 V and the concentration of PBS reaches 39.82 mmoL/L, the ECL intensity reaches its maximum. When the detection potential and pH of concentration continue to increase, the ECL intensity begins to decrease.

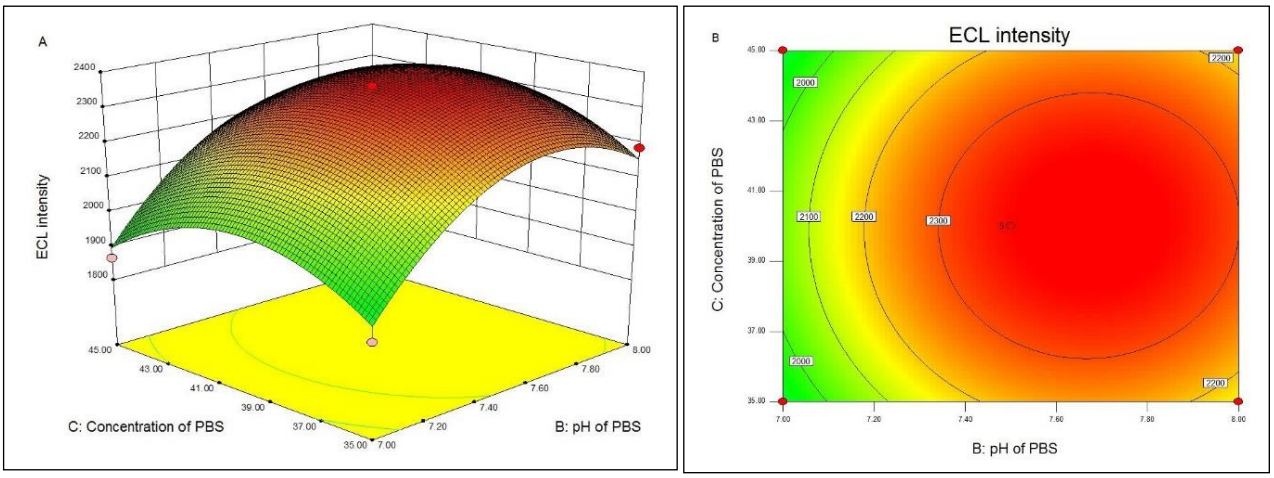

Figure 6. Effect of interaction of pH of PBS and concentration of PBS on ECL intensity.

Figure 6 is 3D surface (A) and contour (B) of the effects of the interaction of pH of PBS and concentration of PBS on the ECL intensity. With the increase of pH of PBS and concentration of PBS, the ECL intensity increases. When the pH of PBS reaches 7.69 and the concentration of PBS reaches 39.82 mmoL/L, the ECL intensity reaches its maximum. When the detection potential and pH of

concentration continue to increase, the ECL intensity begins to decrease.

Based on the above statistical results, it can be found that the optimum values of detection potential, pH of PBS and concentration of PBS were 1.18 V, 7.69 and 39.82 mmoL/L, respectively, when the maximum CTL intensity is obtained. It is almost impossible to obtain the optimal operating conditions through single factor experiments, because the number of experiments needed is very large. According to the model, the maximum value of ECL intensity was 2461.8 (a.u.). This is 4.5% higher than the ECL intensity under single factor optimization conditions. It will correspondingly improve the sensitivity of the method.

3.3 Method performances

Under the optimal experimental conditions, i.e. detection potential 1.18 V, 39.82 mmol/L PBS as buffer solution (pH 7.69) and 5 mmol/L $Ru(bpy)_3^{2+}$, there is a good linear relationship between ECL intensity (I) and MCL concentration (C) in the range of 0.02 - 36.56 mg/L. the linear regression equation is I = 875.6 C + 272.8, the correlation coefficient is 0.9991, and the detection limit (3 σ) of the method is 4.0×10^{-3} mg/L. This result is superior to the existing literature work. See Table 3 for details.

Table 3. Comparison of various methods in this study and Literature.

Number	Methods	Linear range (mg/L)	Detection limit (mg/L)	References
1	HPLC-UV	0.2—10	0.06	34
2	HPLC-MS	1.25—200	0.077	39
3	Spectrophotometry	1.5—15	0.51	42
4	Electrochemistry	0.2—33.5	0.04	43
5	Fluorometry	0.01—0.2	0.007	46
6	CE	2—100	0.5	47
7	ECL	0.02—36.56	0.004	This study

Five standard samples of metoclopramide with different concentrations were prepared in the linear range, and the recovery of the method was 97.3% - 102.4% (see Table 4). The results showed that the method is reliable and accurate.

Table 4. Analysis results of metoclopramide in test samples under 1.18 V detection potential, 39.82 mmol/L PBS as buffer solution (pH 7.69) and 5 mmol/L $Ru(bpy)_3^{2+}$.

Number	Actual concentration (mg/L)	Testing concentration (mg/L)	Recovery （%）
1	1.00	0.98	98.0
2	5.00	5.12	102.4
3	15.00	14.60	97.3
4	25.00	24.73	98.9
5	35.00	35.56	101.6

4. CONCLUSION

On the basis of single factor experimental conditions, the interactions of different factors on electrochemiluminescence signals were investigated by response surface methodology, which can increase the sensitivity of method by 4.5%. Finally, a rapid, accurate, sensitive and simple electrochemiluminescence method for metoclopramide determination was established. This method can directly determine the content of metoclopramide in complex system without separation in the absence of other organic amines.

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