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Spectrophotometric Determination of Captopril in Pharmaceutical Sample by Phosphomolybdic Blue

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Abstract. In the sulfuric acid medium, ammonium molybdophosphate can be reduced to phosphomolybdic blue by hydrosulfuryl(-SH) in captopril molecule, and the content of captopril can be obtained based on the absorbance of the phosphomolybdic blue. A novel method for the spectrophotometric determination of captopril by phosphomolybdic blue has been established. The various effect factors on the spectrophotometric determination of captopril by silicomolybdenum blue are investigated in detail. The results show that the maximum absorption wavelength of chromogenic system is 711.8 nm. Beer's law is obeyed between the concentration of captopril and the absorbance in the range of 0.08024~0.2728 mg/mL, the linear regression equation is A= -0.2635+5.0469C (mg/mL), with the linear correlation coefficient is 0.9991. This proposed method has been applied to determinate of captopril in captopril tablets, and the results agree well with pharmacopoeial method.

1. Introduction

Captopril (CAP,the molecular structure is shown in Figure 1) is is a kind of hypotensive, which is widely used in the clinical treatment of hypertension, coronary disease, cardiac insufficiency and so on. But captopril may cause side effects such as cough, liver injury, leucocytopenia, etc. Thus, the study for the determination of captopril is of great importance and significance for life science.

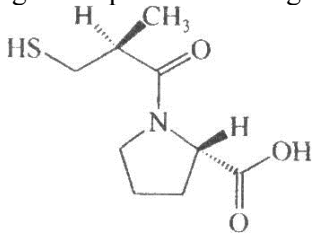

Figure 1. The molecular structure of captopril

So far, there are many methods have been reported for the determination of captopril, such as chemiluminescence[1-2], capillary electrophoresis[3], sequential injection spectrophotometry[4], voltammetry [5-7], liquid chromatography[8], etc.

In this paper, a new spectrophotometric method for the determination of captopril by phosphomolybdic blue is studied. Under the optimum conditions, in the sulfuric acid medium, ammonium molybdophosphate can be reduced to phosphomolybdic blue by hydrosulfuryl (-SH) in captopril molecule, according to the relationship between the amount of captopril and the amount of the phosphomolybdic blue, the content of captopril can be determinated indirectly by measuring the

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absorbance of the phosphomolybdic blue. This proposed method has been applied to determinate of captopril in captopril tablets, and the results agree well with pharmacopoeial method.

2. Experimental

2.1 Equipment and reagents

UV-2401 UV-visible spectrophotometer (The Shimadzu Corporation,japan); 723S spectrophotometer (Shanghai Precision & Scientific Instrument Co,.Ltd).

Captopril standard solution: 2.006 $mg \cdot mL^{-1}$. KH_2PO_4 solution: 2%. Ammonium molybdate(AM) solution: 0.1200g/mL. H_2SO_4 solution: 0.1980 $mol \cdot L^{-1}$. Bi^{3+} solution: 1%. NH_3-NH_4Cl buffer solution: pH=10.

All reagents used were of analytical reagent grade and all solutions were prepared with bidistilled water.

2.2 Method

0.1200 $g \cdot mL^{-1}$ ammonium molybdate solution (4.00 mL), bidistilled water (5.00 mL), 2% KH_2PO_4 solution (0.60 mL), 0.1980 $mol \cdot L^{-1}$ H_2SO_4 solution(0.30 mL), 1% Bi^{3+} solution (0.20 mL) and appropriate amount captopril solution were added into a 25 mL volumetric flask, the solution was mixed well. Aftering the solution reacted for 60 min at 90℃ in water both and cooled back to room temperature, then pH=10 NH_3-NH_4Cl buffer solution (1.00 mL) were added, the solution was diluted to the mark and mixed well. The absorbance was measured at 711.8 nm against the reagent blank after placing 20 min.

3. Results and discussion

3.1. Absorption spectrum

In the range of 550～850nm, the absorption spectrum of phosphomolybdic blue is shown in Figure 2. It can be seen that the maximum absorption wavelength of phosphomolybdic blue formed from ammonium molybdophosphate and captopril in system was 711.8 nm. So, 711.8 nm is selected.

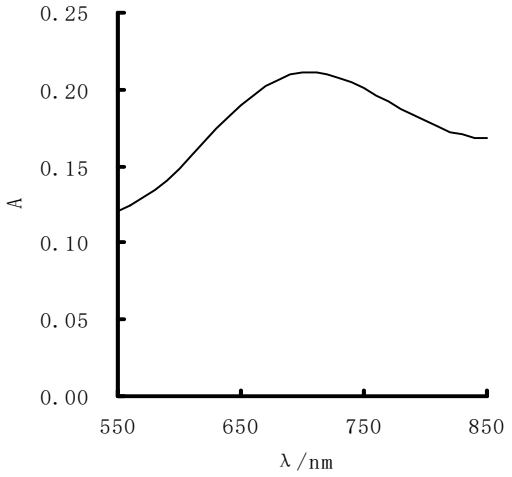

Figure 2. Absorption spectrum
MA:5.00mL;KH_2PO_4:0.50mL;H_2SO_4:0.50mL;
Bi^{3+}:0.20mL;CAP:3.00mL;reaction temperature:
80℃;reaction time:30 min; placing time:10 min;
pH=10 buffer solution:1.00 mL.

3.2. Effects of the reaction temperature, reaction time and placing time

ammonium molybdate solution (5.00 mL), bidistilled water (5.00 mL), KH_2PO_4 solution(0.50 mL), H_2SO_4 solution (0.50 mL), Bi^{3+} solution (0.20 mL), captopril standard solution (3.00 mL) and pH=10 NH_3-NH_4Cl buffer solution (1.00 mL) are applied to the proposed method. The absorbance of different reaction temperature (30,35,40,45,50,55,60,65,70,75,80,85,90,95,100℃) is measured aftering the mixture react for 30 min and after placing 20 min. The results show that the absorbance reaches maximum value and remains constant when the temperature is 90～95℃. Hence, 90℃ is chosen.

Other conditions remain unchanged, the absorbance of different reaction time (10,15,20,25,30,35, 40,45,50,55,60,65,70 min) is measured at 90℃. It is found that the absorbance of solution reaches maximum value and does not change when the reaction time is 60～70 min. So, 60 min is employed.

Other conditions remain unchanged, when reaction temperature is 90℃ and reaction time is 60 min, the effect of the placing time (5,10,15,20,25,30,35 min) is studied. The results show that the absorbance reaches maximum value and remains constant when the placing time is 15～25 min. Hence, 20 min is selected.

3.3. Effect of the dosage of Bi^{3+}

Bi^{3+} has catalysis, it can promote the reaction of ammonium molybdophosphate and captopril. Keeping the dosage of ammonium molybdate solution at 1.00 mL, bidistilled water at 1.00 mL, KH_2PO_4 solution at 0.50 mL, H_2SO_4 solution at 0.50 mL, captopril standard solution at 3.00 mL, pH=10 NH_3-NH_4Cl buffer solution at 1.00 mL, reaction temperature is 90℃, reaction time is 60 min and placing time is 20 min, the effect of the dosage of Bi^{3+} on absorbance is discussed. The results show that the absorbance reaches maximum value when the dosage of Bi^{3+} was 0.20 mL, this clearly means that the catalytic activity of Bi^{3+} reaches the maximum. So, 0.20 mL of Bi^{3+} is chosen.

3.4. Effect of the dosage of H_2SO_4

The effect of the dosage of H_2SO_4 on absorbance is studied(Figure 3). It can be seen from Figure 3 that the absorbance of solution reaches maximum value and keeps constant when the dosage of H_2SO_4 is 0.30～0.40 mL. So, 0.20 mL of the dosage of H_2SO_4 has been selected.

3.5. Effect of the dosage of KH_2PO_4

The effect of the dosage of H_2SO_4 on absorbance is studied(Figure 4). The results show that the absorbance reaches its maximum value and remains constant when the dosage of KH_2PO_4 is 0.40 mL～0.80 mL. Hence, 0.60 mL of KH_2PO_4 is chosen.

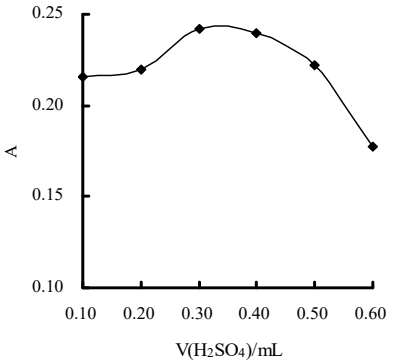

Figure 3. Effect of the dosage of H_2SO_4
MA:5.00mL; KH_2PO_4:0.50 mL; Bi^{3+}:0.20 mL;
CAP:1.50 mL; reaction temperature:90℃;
reaction time:60 min; placing time:20 min;
pH=10 buffer solution:1.00 mL.

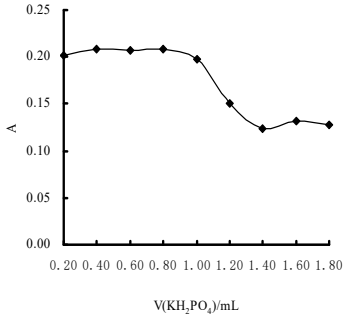

Figure 4. Effect of the dosage of KH_2PO_4
MA:5.00 mL; H_2SO_4:0.30 mL; Bi^{3+}:0.20 mL;
CAP:1.50mL;reaction temperature:90℃;
reaction time:60 min; placing time:20 min;
pH=10 buffer solution:1.00 mL.

3.6. Effect of the dosage of ammonium molybdate(AM)

The dosage of ammonium molybdate is regarded as an important factor on the formation of phosphomolybdic blue. The effect of AM on absorbance can be seen in Figure 5. As shown in Figure 5, with the increase of AM dosage, the absorbance increase. The absorbance reaches its maximum value and and it does not change any further when the amount of AM is 4.00 mL～5.00 mL. So, 4.00 mL of AM has been chosen.

3.7. Calibration curve

Under the selected optimum conditions, a series of standard solutions of captopril is prepared, then the absorbance is measured at 711.8 nm against the reagent blank. A linear relationship between absorbance (A) and the concentration (C) of captopril is obtained in the range of 0.08024~0.2728 mg/mL (Figure 6). The linear regression equation is A=-0.2623+5.0469C (mg/mL), with a correlation coefficient of 0.9991.

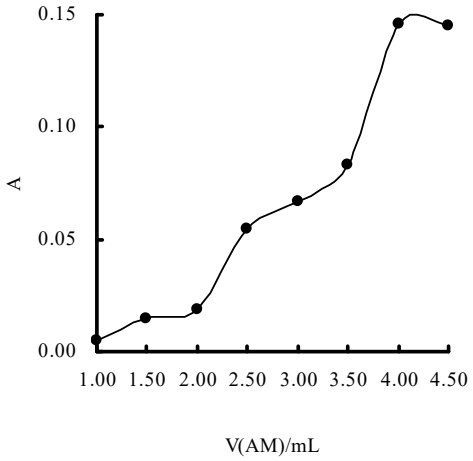

Figure 5. Effect of the dosage of AM
KH_2PO_4:0.50mL; H_2SO_4:0.30mL; Bi^{3+}:0.20mL; CAP:1.50 mL; reaction temperature:90℃; reaction time:60 min; placing time:20 min; pH=10 buffer solution:1.00 mL.

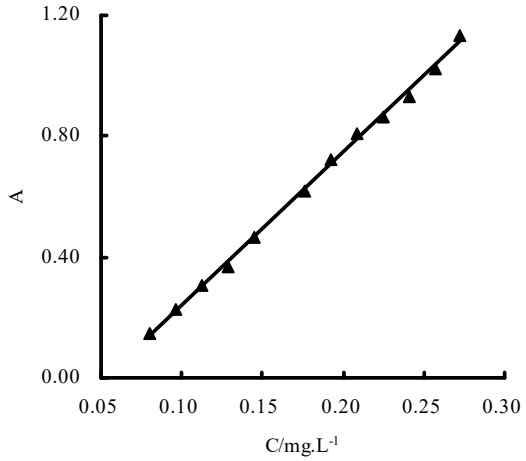

Figure 6. Calibration curve
MA:4.00mL; KH_2PO_4:0.60mL; H_2SO_4:0.30mL; Bi^{3+}:0.20 mL; reaction temperature:90℃; reaction time:60 min; placing time:20 min; pH=10 buffer solution:1.00 mL.

3.8. Determination of captopril in pharmaceutical sample

Forty tablets of captopril tablet are weighed 3.4523g, round and blended. 3.0070g g powder of captopril is weighed precisely and dissolved in bidistilled water and is transferred into a 250 mL volumetric flask, the solution is diluted to the mark with bidistilled water and mixed well, this is a sample solution, stand-by.

Sample solution (20.00 mL) is diluted to 100 mL with bidistilled water and mixed well. The solution is preserved at 4℃, shilding from light. Based on the experimental method, the prepared solution of captopril sample is determined. The content of captopril in captopril tablet can be obtained. Meanwhile, the recovery tests of standard addition are performed. The results obtained is compared with those obtain by pharmacopoeia method, as show in Table 1.

Table 1. The determination result of captopril in captopril tablet n = 5

Sample	Proposed method ($mg \cdot tablet^{-1}$)	RSD (%)	Pharmacopoeia method[9] ($mg \cdot tablet^{-1}$)	Added ($\mu g \cdot mL^{-1}$)	Recovered ($\mu g \cdot mL^{-1}$)	Recovery (%)
Captopril tablet	25.58	0.4	24.32	32.10 80.24	32.02 79.77	99.75 99.41

Table 1 shows that the content of captopril in captopril tablet is 25.58 $mg \cdot tablet^{-1}$ by this proposed method, agreed well with 24.32 $mg \cdot tablet^{-1}$ obtain by pharmacopoeial method.

4. Conclusion

A novel method for the spectrophotometric determination of captopril by phosphomolybdic blue has been established. The proposed method has been successfully applied to the determination of captopril in pharmaceutical sample with satisfactory results. This proposed method has the advantages of simply, rapidness, convenience, accuracy and so on. It is obvious that the determination of captopril by phosphomolybdic blue spectrophotometry has certain practical significance and foreground of application.

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