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Spectrophotometric Determination of Magnesium(II) with *m*-Acetylchlorophosphonazo

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The optimum conditions of spectrophotometric determination of Mg(II) with *m*-acetylchlorophosphonazo (CPA*m*A) were studied. In a medium of pH 10.5 NH_3-NH_4Cl, the maximum absorption peak of Mg (II)-CPA*m*A complex is located at 610 nm. At this wavelength, Beer's law is obeyed over the range of 0-1.5 μg/mL of Mg(II) and the apparent molar absorptivity of the complex is 2.99×10^4 L mol^{-1} cm^{-1}. Detection limit of the method is 7.71 ng/mL. When the proposed method was used to determine magnesium in serums, the determined results were in good agreement with those obtained by atomic absorption spectrometry. The recovery of the standard addition for the determination of serum sample by the present method was 97.59-99.71 % and the relative standard deviation for eleven replicate determinations was 1.36-3.47 %. The analytical results were satisfactory.

Key Words: *m*-Acetylchlorophosphonazo, Magnesium, Spectrophotometry, Serum.

INTRODUCTION

Magnesium(II) is one kind of indispensable elements that participate in the normal life activity and metabolic process of biological body, affecting the transfers of potassium ions and calcium ions, participating in energetic metabolism and the synthesis of protein and nucleic acid. Magnesium can activate many kinds of enzymes *in vivo*, adjusting the activities of nerve, muscle and nerve centre system, ensuring the normal constriction of cardiac muscle, almost participating in all metabolism process[1,2]. Studies showed that human body lacks magnesium, which can arouse many diseases hypertension, diabetes, coronary heart disease, myocardial infarct, *etc*.[3]. Therefore,study and establishment of the analytical method for the determination of magnesium have an important significance.

Spectrophotometric determination of magnesium has a greater practical value due to the advantages of having operation simplicity and low cost instrumentation, *etc*.[4]. Although some spectrophotometric methods for the determination of magnesium have been proposed and 2, 2', 6', 2''-terpyridine[4], 2-(4-sulfophenylazo) chromotropic acid[5], chomeazurol S[6], 4, 5-dibrophenylfluorone[7], dibromonitroarsenazo[8], chlorophosphonazo-I[9], 4-(2-hydroxy-4-nitrophynylazo)-1-phenyl-3-methylpyrazolone[10] have already been explored for the determination of magnesium, selectivity of the methods are still not ideal. Thus,development of the novel spectrophotometric method for determination of magnesium still has important significance. *m*-Acetylchlorophosphonazo (CPA*m*A) is easily soluble in water and has been used in the spectrophotometric determination of protein[11]. This study used CPA*m*A as chromogenic agent and established the optimum reaction conditions for the spectrophotometric determination of magnesium(II). It has been successfully applied to the determination of magnesium(II) in serum samples and satisfactory analytical results were obtained.

EXPERIMENTAL

A 721E spectrophotometer (Shanghai Spectral Instrumentation Corporation, Ltd., China) with 1 cm cells was used in the absorbance measurement. Standard stock solution (1 mg/mL) of Mg^{2+} was prepared by dissolving 0.1658 g MgO in 1 mL of conc. hydrochloric acid and the content was diluted to 100 mL. Then, the working solution (2 μg/mL) was prepared by the suitable dilution of the stock solution. 7.4×10^{-4} mol/L *m*-acetylchlorophosphonazo (CPA*m*A, Shanghai Jinsheng Chemical Corporation, Ltd., China) solution was used. pH 10.5 NH_3-NH_4Cl buffer solution was used to control the acidity of colour reaction. All the reagents used were of analytical pure grade.

In 10 mL calibrated flasks, in turn 6 μg of magnesium (II) working solution, 1.0 mL of 7.4×10^{-4} mol/L CPA*m*A solution and 1.0 mL of pH 10.5 NH_3-NH_4Cl buffer solution were added. Water was added to the above solution to the mark and the solution was shaken. After the solution was set for

5 min, the absorbance of colour solution was determined at 610 nm with 1 cm cells using reagent blank as reference.

RESULTS AND DISCUSSION

Absorption spectra: According to the standard procedure, the absorption curves of reagent blank against water and complex against reagent blank were respectively drawn over the range of 400-800 nm. The results showed (Fig. 1) that the maximum absorption of reagent blank is located at 580 nm and the maximum absorption of complex is located at 610 nm. The wavelength for determination of Mg(II) was selected to be 610 nm.

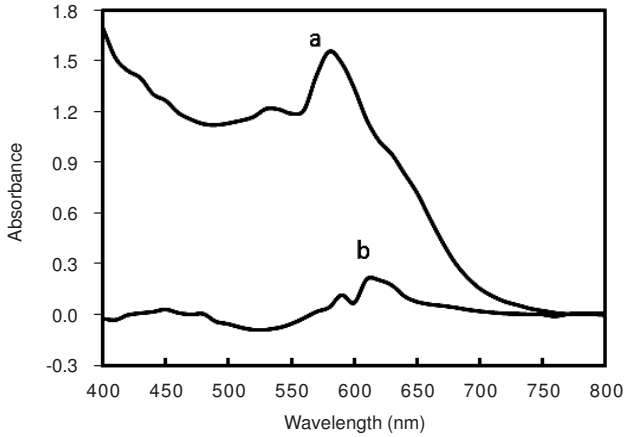

Fig. 1. Absorption spectra: (a) CPA*m*A (against water); (b) complex(against reagent blank). $[Mg^{2+}] = 2.5 \times 10^{-5}$ mol/L; [CPA*m*A] = 7.4×10^{-5} mol/L; pH =10.5

Effect of acidy: The experimental results showed that the CPA*m*A-Mg(II) complex shows colour in basic medium. The absorbance of complex increased as pH increased over the range of 8-10.5. When pH was within 9.8-10.8, the absorbance of complex tended smooth as pH increased. At pH 10.5, the absorbance was a maximum. The absorbance of complex decreased as pH increased within pH 10.8-12. Thus, in this study pH = 10.5 NH_3-NH_4Cl buffer solution was chosen to control the acidity of system.

The effect of buffer solution amount on the absorbance of complex was not great. As the amount of buffer solution increased within 0.2-1.0 mL, the absorbance of complex increased. At 1 mL, the absorbance reached a maximum. The absorbance decreased as the amount of buffer solution increased within 1-2 mL. Therefore, in this study 1 mL of pH = 10.5 NH_3-NH_4Cl buffer solution was selected for further study.

Effect of the amount of CPA*m*A: The experimental results showed that when the added amount of CPApA was 0- 0.5 mL, the absorbance of complex increased with increase in the amount of chromogenic agent. Over the range of 0.5-1.8 mL, the absorbance was maximum and steady. Within 1.8-2.5 mL, the absorbance decreased with increase in the amount of chromogenic agent. 1.0 mL of 7.4×10^{-4} mol·L^{-1} CPA*p*A solution was used in this study. At this time, [CPA*m*A] = 7.4×10^{-5} mol/L.

Effect of surfactant: According to the standard procedure, the effect experiments of cetyltrimethylammonium bromide, tween-80, OP-100 and sodium dodecylsulphate were respectively carried out. The results showed that OP-100 and sodium dodecylsulphate have not effect on the sensitivity of colour reaction and cetyltrimethylammonium bromide and Tween-80 made the sensitivity of colour reaction reduce.

Effect of temperature: According to the standard procedure,the effect of temperature at 4, 20, 30, 40, 50, 60, 70, 80, 90,100 ºC was respectively carried out. The results showed that the CPA*m*A-Mg(II) complex absorbance value was basically unchangeable as the temperature increased. The temperature has basically no effect on the reaction. Thus, the experiment can be made at room temperature.

Complex stability and composition: Under the conditions of present experiment, the complex reaction of Mg(II) and CPA*m*A promptly accomplished. The produced complex could retain stable within 2 h and the variation of absorbance was less than 5 %. The molar ratio of CPA*m*A with Mg(II) in the complex, determined by molar ratio method and equimolar continuous variation method, was 1:1.

Linear range: Suitable amount magnesium(II) working solutions were taken and placed in 10 mL calibrated flasks and colour development was made according to the standard procedure for the determination of absorbance. The results showed that over a range of 0-15 μg/10 mL for Mg(II) Beer's law is obeyed and the linear range of working curve is: A = 0.1531 C(C:μg/mL) + 0.0069, with a correlation coefficient of r = 0.9991. From the working curve it was calculated that at 610 nm the apparent molar absorptivity of complex is 2.99×10^4 L·mol^{-1}·cm^{-1}. For eleven parallel determinations of 0.24 μg/mL Mg(II) working solution, the relative standard deviation of method was calculated to be 1.03 %. For eleven parallel determinations of reagent blank, the detection limit of method was calculated to be 7.71 ng / mL according to 3S/K method (S is the standard deviation of eleven blank experiments, K is the slope of regression equation).

Effect of co-existing ions: Under the optimum experimental conditions, the effect experiments of co-existing ions were made. In 10 mL volumetric flask for the determination of 6 μg of Mg(II) using CPA*m*A as the chromogenic agent, the following amounts (m/m) of co-exiting ions did not cause interference within a relative error of ± 5 % : SiO_3^{2-}, F^- (150); SO_4^{2-} (100); NO_2^- (60); NO_3^-, Br^-, BrO_3^- (50); MoO_4^{2-} (25); PO_4^{3-} (20); $S_2O_7^{2-}$ (8). Zn^{2+}, Pb^{2+}, Co^{2+}, Mn^{2+}, Ag^+ (5); Cr (VI), Bi^{3+},Fe^{2+}, Ni^{2+}, Cu^{2+}, Ba^{2+}, Li^+, MnO_4^-, VO_3^-, I^- (2); Fe^{3+} (2, 10[a]), Al^{3+} (2, 10[a]); Cr^{3+}, CH_3COO^- (1); Ca^{2+} (0.05, 0.2[a]). Salicylic acid, ascorbic acid (500); glucose, citric acid (200); bovine serum albumin, cysteine (40); lysine (30); bovine hemoglobin (20); phenylalanine, glutamic acid, leucine (10) (a : 2.0 mL of 0.5 mg/mL EDTA-Na_2 were added).

Analytical application: 0.2 mL of serum sample were accurately taken and placed in a beaker. 2 mL of concentrated nitric acid and 4 mL of concentrated hydrogen peroxide were added, the content was evaporated to near dryness, the left-over substance was dissolved by 2 mL of 0.1 mol /L hydrochloric acid and then transferred to a 10 mL comparison tube. It was adjusted to neutrality by 0.2 mol/L sodium hydroxide and then 2.0 mL of 0.5 m g/mL EDTA-Na_2 were added. The remainder was the same as the standard procedure for the

determination of magnesium (Table-1). It can be seen that the results determined by the present method can be very well contrasted with those by atomic absorption spectrometry. The relative standard deviation of five determinations was 1.36-3.47 % and the recovery was between 97.59-99.71 %. The analytical results were quite satisfactory.

TABLE-1
ANALYTICAL RESULTS OF SAMPLE

Sample	Found (n = 5, %)	Relative standard deviation (%)	Recovery (%)	Atomic absorption spectrometric contrast method (%)
No. 1	18.11	1.36	97.59	18.11
No. 2	20.56	3.47	99.71	20.57

Conclusion

This paper established a novel method for the spectrophotometric determination of magnesium with *m*-acetylchlorophosphonazo and was successfully applied to determine magnesium(II) in serum samples. The maximum absorption peak of the Mg(II) complex is located at 610 nm, $\varepsilon_{610\,nm} = 2.99 \times 10^4$ L·mol^{-1}·cm^{-1}. The linear range for the determination of Mg(II) is 0-1.5 μg/mL and the linear regression equation of working curve is A = 0.1531 C(C:μg/mL) + 0.0069, with a correlated coefficient of r = 0.9991. Detection limit of the method is 7.71 ng/mL.

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