



Research Article

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## Color reaction of *m*-acetylchlorophosphonazo and nickel(II)

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### ABSTRACT

In pH 6.8 triacid (phosphoric acid, acetic acid, boric acid)-NaOH buffer medium, nickel (II) and *m*-acetyl-chlorophosphonazo (CPAmA) form a 1 : 3 blue complex. At the maximum absorption wavelength of 610 nm, Beer's law is obeyed over the range of 0.1 ~ 4.0  $\mu\text{g} / \text{mL}$  for nickel content and the apparent molar absorption coefficient of method is  $\epsilon_{610\text{nm}} = 1.13 \times 10^4 \text{ L mol}^{-1}\text{cm}^{-1}$ . This method has been applied to the determination of nickel in kelp.

**Keywords:** *m*-acetylchlorophosphonazo, nickel, color reaction.

### INTRODUCTION

Nickel is an essential trace element for human body. An appropriate amount of nickel can promote the body's normal physiological function. Nickel can make coronary artery contraction and expansion, inhibition of coronary artery blood pressure, therefore, can alleviate myocardial hypoxia response. Nickel decreased in liver disease and anemia, and nickel was increased in cerebrovascular disease, acute myocardial infarction and other diseases [1]. In the surrounding environment, source of nickel is wide, such as humans can absorb nickel through the food chain, therefore, to study the method for determination of nickel in food has a very important significance.

Spectrophotometric method has the advantages of simple operation, high accuracy, instrument and low analysis cost. In recent years, in the determination of trace nickel it is still studied. Some color systems for the determination of nickel have been put forward, 2-[2'-(5-nitro-pyridylazo)]-1, 8-dihydroxynaphthalene-3, 6-disulfonic acid [2], 2, 2-bi-3,3'-disulphodiazoaminoazo-benzene propane [3] and 4-(6-methoxy-8-quinolylazo)-resorcinol [4] have been used for the discussion of nickel determination chromogenic agent. But these reagents were used for the determination of nickel, the selectivity is still not ideal. Therefore, development of a new method for the determination of nickel still has practical significance and value. *m*-Acetylchlorophosphonazo (CPAmA), whose structure is shown in Figure 1, has been used in the spectrophotometric determination of rare earth elements [5], iron [6], protein [7]. To the best of our knowledge, no study on color reaction of CPAmA and  $\text{Ni}^{2+}$  is reported. In this paper color reaction of the reagent with nickel (II) is studied, the optimal experimental conditions for the determination of nickel are established and the properties of chromogenic system are studied. The present method has been successfully applied to the determination of nickel in food kelp.

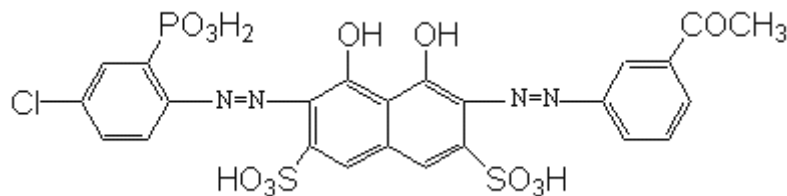


Fig.1: Structure of CPAmA

## EXPERIMENTAL SECTION

### Reagents and apparatus

All reagents used in the experiment were of analytical-reagent grade and the water was distilled throughout the study. The stock standard solution of nickel, 1 mg / mL, was prepared by dissolving 0.4784 g of  $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$  in an appropriate amount of water containing 2 mL of  $\text{H}_2\text{SO}_4$  (1 + 1, V/V) and then diluted to 100 mL with water. The working standard solution of nickel(II), 10  $\mu\text{g}$  / mL, was prepared by appropriate dilution of the standard solution with water. A 0.5  $\text{g L}^{-1}$  *m*-acetyl-chlorophosphonazo (CPAmA) solution was prepared in water. Buffer solution (pH = 6.8): 50 mL of 0.02 mol / L NaOH solution was added to 100 mL of three acids (phosphoric acid, acetic acid, boric acid) mixed solution whose concentrations were all 0.04 mol/L for each acid.

A 722 spectrophotometer (Shanghai Prism Light Technology Corporation, Ltd., China) with 1 cm cells was used for all absorbance measurements.

### General procedure

A definite amount (for conditional experiment: 20  $\mu\text{g}$ ) of  $\text{Ni}^{2+}$  was placed in a 10-mL calibrated flask, and 1.5 mL of CPAmA chromogenic agent solution and 2.0 mL of buffer solution were added. The above solution was diluted to the mark with water. After 10 min, the absorbance was measured on a spectrophotometer at 644 nm in a 1 cm cell against a reagent blank prepared by the same procedure.

## RESULTS AND DISCUSSION

### Determination wavelength

In pH = 6.8 three acids-NaOH medium, nickel (II) can form a purple blue complex with CPAmA. Its maximum absorbance is at 610 nm (Fig. 2), while that of CPAmA is at 560 nm. In order to obtain maximum sensitivity, in the following study 610 nm was selected as the determination wavelength.

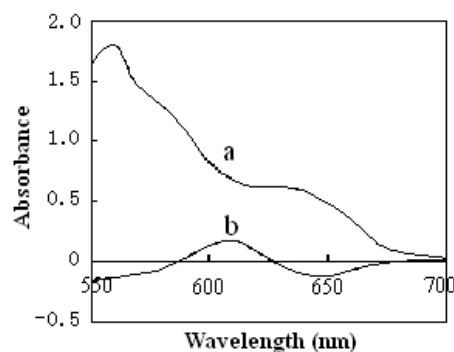


Fig.2: Absorption spectra

(a) Reagent blank (against water); (b) complex (against reagent blank);  $[\text{Ni}^{2+}] = 3.41 \times 10^{-5}$  mol / L; pH = 6.8

### Effect of acidity

The experimental results indicated that a maximum absorbance could be obtained over the pH 6.4-7.2. In the following experiment, pH 6.8 was chosen. When the amount of buffer solution was 1.8-2.3 mL, absorbance was a maximum. In the experiment, 2.0 mL was selected.

### Effect of CPAmA amount

The experiments showed that over the range of 0 — 1.0 mL the absorbance gradually raised with increasing the amount of CPAmA. When the amount of CPAmA was 1.0 — 2.0 mL, the absorbance was a maximum. In the present experiment 1.5 mL of 0.5 g/L CPAmA solution was chosen.

### Complex composition and its stability

The composition was evaluated by the molar ratio method and continuous variation method. Both showed that the molar ratio of Ni(II) to CPAmA is 1: 3.

Under the optimum experimental conditions, for the determination of 2.0 µg / mL Ni<sup>2+</sup> the results showed that speed of the color reaction was very fast at a room temperature and absorbance of the color reaction could instantly reach maximum and invariable and the complex formed could be stable for 2 h at least.

### Analytical characteristics

The calibration graph was constructed according to the general procedure under the optimum conditions. The results showed that Beer's law was obeyed over the 0.1 — 4.0 µg / mL of nickel(II). Its regression equation was:  $A = 0.1835 C (\mu\text{g} / \text{mL}) + 0.0023$ , with a correlation coefficient of 0.9997. The apparent molar absorptivity was calculated to be  $\epsilon_{610\text{nm}} = 1.13 \times 10^4 \text{ L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ .

Eleven replicate analyses of a test solution containing 3.0 µg / mL Ni<sup>2+</sup> by the general procedure gave a relative standard deviation of 1.17 %, indicating that the precision of method was good and the reproducibility of the determination experiments was nice. For eleven determinations of a blank reagent, the detection limit of method 3S / K (S is standard deviation and K is slope of the working curve) obtained was 0.061 µg / mL.

### Effect of foreign ions

Under the optimum conditions, the effects of various foreign substances on the determination of 20µg Ni<sup>2+</sup> were studied. Tolerable levels (±5% maximum error) of foreign substances on the determination of nickel are as follows (Multiple, W/W): F<sup>-</sup>, Cl<sup>-</sup>, Br<sup>-</sup>, CH<sub>3</sub>COO<sup>-</sup>, SiO<sub>3</sub><sup>2-</sup>, SO<sub>4</sub><sup>2-</sup>, PO<sub>4</sub><sup>3-</sup> (1500); B(III), I<sup>-</sup> (400); Ag<sup>+</sup>, Cd<sup>2+</sup> (200); Mg<sup>2+</sup> (50); Cr(VI) (5); Cu<sup>2+</sup>, Al<sup>3+</sup>, Fe<sup>3+</sup>, Y<sup>3+</sup>, Zr(IV), Ce(IV), Mo(VI) (4); Zn<sup>2+</sup>, Fe<sup>2+</sup>, Co<sup>2+</sup>, Mn<sup>2+</sup>, Ti(IV), W(VI), MnO<sub>4</sub><sup>-</sup> (2); Ca<sup>2+</sup>, Sr<sup>2+</sup>, Ba<sup>2+</sup>, La<sup>3+</sup> (0.1); ascorbic acid, ammonium tartrate (8); oxalic acid (5). The selectivity of the method is better than those in the literature [2-4] with respect to Cd<sup>2+</sup> and Ti<sup>4+</sup>.

### Sample analysis

50 g of kelp sample was accurately weighed, desiccated in an oven at 110°C for 4 h. The sample was taken out, carbonized on an electric cooker by heating, and then placed in a muffle oven at 500°C for ashing for 3 h. The sample was taken out. After it was cooled, the ash was dissolved by 5 mL of 2 mol / L hydrochloric acid. It was heated on an electric cooker until it was evaporated to near dryness. After a small amount of water was used for dissolution, the contents were diluted to 10-mL. An appropriate amount of the above-stated solution was taken and placed in a 10-mL calibrated flask and nickel was determined according to the general procedure. At the same time a recovery experiment of the sample was made out and a contrast experiment was done with atomic absorption spectrometry. The determined results of nickel in the sample are listed in Table 1.

Table 1: Analytical results of samples

Sample	Average found (n = 9, µg / g)	Relative standard deviation (%)	Added (µg / g)	Recovered (µg / g)	Recovery (%)	Atomic absorption contrast spectrometry contrast method (µg / g)
No.1	4.23	0.99	5.000	4.997	99.94	4.22
No.2	2.11	1.9	5.000	5.001	100.02	2.13

## CONCLUSION

This paper established a new spectrophotometric method using *m*-acetyl-chlorophosphonazo for the determination of nickel. Nickel content in the range of 0.1 ~ 4.0 µg / mL obeys Beer's law and the apparent molar absorption coefficient of method is  $\epsilon_{610\text{nm}} = 1.13 \times 10^4 \text{ L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ . This method has been successfully applied to the determination of nickel in kelp. The operation of method is simple, fast.

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