



Research Article

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Determination of nickel in tea with arsenazo-III by spectrophotometry

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ABSTRACT

Using arsenazo-III as chromogenic reagent, a spectrophotometric method for the determination of nickel was developed. The maximum absorption wavelength of nickel complex is 610 nm. Beer's law is conformed over the range of 0.2 ~ 4.0 $\mu\text{g} / \text{mL}$ for nickel. The apparent molar absorption coefficient of method is $\epsilon_{540\text{ nm}} = 1.76 \times 10^4 \text{ L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$ and detection limit is 0.093 $\mu\text{g} / \text{mL}$. The present method has been already applied to the determination of nickel in tea and method recovery is between 99.7% ~ 100.1%.

Keywords: Nickel, spectrophotometry, arsenazo-III, tea

INTRODUCTION

Nickel is a relatively abundant element in the earth's crust. It is also one of the 14 microamount elements that the human body must need. It is involved the metabolism of certain enzymes in the body, stimulates hematopoiesis function, promotes the regeneration of red blood cells and can treat anemia and liver cirrhosis. It has a buffer action on asthma, cardiac and pulmonary dysfunction and pulmonary heart disease and plays a certain role to maintain physiological function. However, in the human body the absorbing too high nickel will hurt the myocardium, lung, liver and kidney, and even results in carcinogenic [1]. Humans can absorb nickel by drinking tea water drinks etc, therefore, it has an important practical significance to study and establish a method for the determination of nickel in tea samples.

Although the research discussions have been reported on some spectrophotometric methods for the determination of nickel, such as 2, 2-bi-3,3'-disulphodiazaminoazo- benzenylpropane [2], *p*-nitrodiazaminoazabenzene [3], the selectivity of these methods are still not ideal and a new method for the determination of nickel still needs to be established. Arsenazo-III (abbreviated as ASA-III, known as 2,7-bis(2-arsenophenylazo)-1,8-dihydroxy naphthalene-3,6-disulfonic acid) as a chromogenic reagent of spectrophotometric analysis has been used for the determination of rare earths [4], calcium [5], protein [6]. To the best of our knowledge, no study report on the spectrophotometric determination of nickel in tea is seen. In this paper, ASA-III is used as chromogenic agent and a chromogenic reaction of the reagent with nickel(II) is studied and successfully used for the determination of nickel in tea with satisfactory results. This method has the advantages of low instrument cost and simple operation.

EXPERIMENTAL SECTION

Reagents and apparatus

All reagents used in the experiment were of analytical-reagent grade and the water was distilled. The stock standard solution of nickel, 1 mg / mL, was prepared by dissolving 0.4784 g of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ (Chinese Pharmaceutical Group, China) in an appropriate amount of water containing 2 mL of H_2SO_4 (1 + 1, V/V, (Chinese Pharmaceutical Group, China) and then diluted to 100 mL with water. The working standard solution of nickel(II), 10 $\mu\text{g} / \text{mL}$, was prepared

by appropriate dilution of the standard solution with water. A 1 g/L arsenazo-III (ASA-III, Shanghai Research Institute of Reagent, China) solution was prepared in water. pH = 5.5 acetic acid - sodium acetate (Chinese Pharmaceutical Group, China) buffer solution was used for control of the reaction acidity.

A 722S spectrophotometer (Shanghai Linggunag Technique Co., Ltd., China) equipped with 1.0 cm cell was used for measuring the absorbance.

Recommended procedure

An appropriate amount of Ni(II) working solution (for the conditional experiments, 15 μg) was transferred into a 10-mL calibrated flask, and 2.0 mL of arsenazo-III solution, 1.5 mL of buffer solution were consecutively added. Water was used to dilute to the mark. After 2 min, the absorbance of the colored solution was measured at 610 nm against a reagent blank using 1 cm cells.

RESULTS AND DISCUSSION

Absorption spectra

Arsenazo-III can form a violet complex with nickel (II) in pH 6.0 HAc-NaAc medium. The maximum absorption wavelength of the reagent blank against water is at 540 nm, while the complex against reagent blank has maximum absorbance at 610 nm (Fig.1). Therefore, the measurements of the complex were carried out at 610 nm.

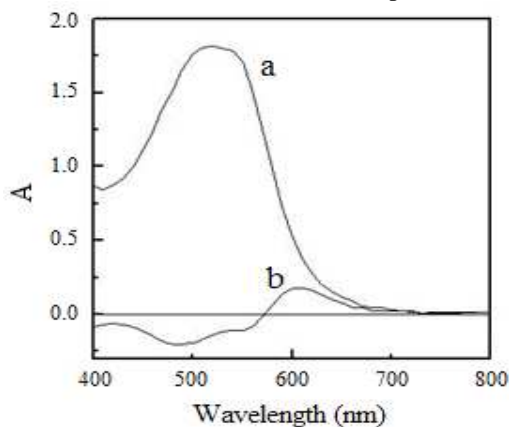


Fig. 1: Absorption spectra

(a) Reagent blank (against water); (b) complex (against reagent blank); $[\text{Ni}^{2+}] = 2.6 \times 10^{-5} \text{ mol/L}$; pH = 5.5

Optimum determination conditions

The results of acidity effect indicated that a maximum absorbance could be obtained under the condition of pH 5.0 — 6.0. Outside the range, the absorbance was lower. pH 5.5 was chosen. When the amount of buffer solution was 1.0 — 2.0 mL, the absorbance was a maximum. In the experiment, 1.5 mL HAc-NaAc buffer solution was chosen as the reaction medium.

For the determination of 1.5 $\mu\text{g/mL}$ of nickel (II), the addition of 1.8 — 2.2 mL of 1 g/L ASA-III chromogenic solution produced a maximum absorbance. Outside the range, the absorbance was lower. For all subsequent determinations, 2.0 mL of 1 g/L ASA-III solution was added.

System properties

Stability and composition of complex

After arsenazo-III and nickel (II) was mixed, a blue complex immediately formed. In this study determination of absorbance was made after various solutions were added for 2 min. The complex produced can be stable for 2 h. Molar ratio method and equal molar continuous variation method were respectively employed to determine the composition ratio of complex to be Ni (II) : chromogenic reagent = 1 : 3.

Working curve

The experimental results showed that between a range of 0.2 ~ 4.0 $\mu\text{g/mL}$ of Ni(II) and the absorbance a good linear relationship is presented and its regression equation is: $A = 0.0413 C (\mu\text{g/mL}) + 0.0061$, with a correlation coefficient of 0.9995. At 610 nm, the apparent molar absorptivity of method is $\epsilon = 1.76 \times 10^4 \text{ L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$. For eleven parallel determinations of 1.5 $\mu\text{g/mL}$ Ni(II), the relative standard deviation of method was calculated to be 1.11%. The detection limit of method was calculated to be 0.13 $\mu\text{g/mL}$ according to $3S/K$ method (S is the

standard deviation of eleven blank experiments, K is the slope of working curve) .

Effect of co-existing substances

The selectivity of the method was tested by studying the effect of various ions on the determination of 1.5 µg/mL of Ni²⁺. The tolerance limits were taken for a maximum error of ±5%. The interference effect of the tested ions are as follows(weight ratio): PO₄³⁻ (50), Al³⁺, I⁻ (10), Cu²⁺(5), Ca²⁺, Mg²⁺(2), Zn²⁺, W(VI) (0.6), Mo(VI) (0.5), Ag⁺, Ce(IV), Cr(VI), F⁻, CH₃COO⁻ (0.4), SiO₃²⁻ (0.2), Fe²⁺, Cd²⁺, B(III) , Y³⁺, Ti⁴⁺, Zr⁴⁺, Cl⁻, Br⁻ (0.1), Mn²⁺, MnO₄⁻ (0.05), Co²⁺, Fe³⁺ (0.01), ascorbic acid, ammonium tartrate, hydroxylamine hydrochloride, oxalic acid (20). Compared with the literature [2, 3], the allowed amounts of ascorbic acid, ammonium tartrate, hydroxylamine hydrochloride, oxalic acid are larger.

Sample analysis

10 g of tea leaf was accurately weighed, desiccated in an oven at 110°C for 2 h, placed in a muffle oven at 500°C for ashing for 2 h, and then taken out to be cooled. To the tea leaf ash, 5 mL of concentrated nitric acid was added and heated on an electric cooker until it was completely dissolved. The contents was evaporated to near dryness, then dissolved in water and transferred to a 25-mL volumetric flask and diluted to the mark with water. 2.0 mL of the solution was taken and nickel content was determined according to the general procedure. Meanwhile, atomic absorption spectrometry was used for contrast experiment and recovery experiment was done. The determined results are seen in Table 1.

Table 1 Analytical results of samples

Sample	Found (n = 11) (µg / g)	Relative standard deviation (%)	Added (µg/ g)	Recovered (µg / g)	Recovery (%)	Atomic absorption contrast spectrometry (µg / g)
No.1	30.33	1.9	2.000	2.002	100.1	30.33
No.2	11.11	1.3	2.000	1.994	99.7	11.10

CONCLUSION

In this paper, optimum experimental conditions for the spectrophotometric determination of nickel have been developed with arsenazo-III as chromogenic agent, the linear range of method for determining nickel is 0.2 ~ 4.0 µg / mL and the apparent molar absorption coefficient is $\epsilon_{610\text{ nm}} = 1.76 \times 10^4 \text{ L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$. It has been satisfactorily applied to the determination of nickel in teas.

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