

NOTE

Colour Reaction of Barium(II) with *p*-Acetylarsono

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The optimum colour conditions of *p*-acetylarsono with barium(II) were established. *p*-Acetylarsono reacted with barium(II) in pH 10.4 NH₃-NH₄Cl buffer solution to form a blue complex with a ratio of 2:1. At the maximum absorption wavelength 664 nm of the complex, Beer's law was accorded in the range of 0.50-6.0 µg/mL of barium(II) amount. The apparent molar photoabsorption coefficient of the method was $\epsilon_{644\text{ nm}} = 9.84 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$. Detection limit was 0.17 µg/mL. The present method has been satisfactorily used in the determination of barium content in water sample.

Key Words: Barium, Spectrophotometry, *p*-Acetylarsono, Water sample.

Barium is a toxic element and its soluble barium salts have greater toxicity. As environmental pollution increasingly aggravates, water quality, soil and air have been come in for prodigious damage. Barium has been listed in detection item of some environmental samples. Thus, determination of barium content in some environmental samples has an important significance.

For determination of barium there are atomic absorption spectrometry, inductively coupled plasma-atomic emission spectrometry, X-ray fluorescence method, spectrophotometry, *etc.*^{1,2}. Compared with other methods, as spectrophotometry has the advantages of operation simplicity, cheap instrumentation price, *etc.*, determination of barium has higher practical value³⁻⁶. However, most selectivity of the spectrophotometric system of determination of barium is not ideal. Therefore, establishment of a new spectrophotometric system of determination of barium has an important meaning.

p-Acetylarsono (ASApA) was ever used for the determination of rare earths⁷. Its structural formula is as follows (Fig. 1):

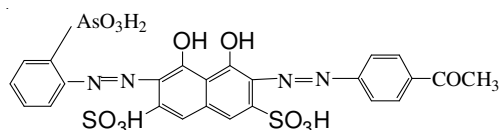


Fig. 1. Structural formula of ASApA

In this paper the reaction conditions of spectrophotometric determination of barium(II) using ASApA and its application

were studied. The present procedure was satisfactorily used in the determination of barium content in lake water. Operation of the method is simple, convenient and fast.

Absorbances were measured on a 722S spectrophotometer (Shanghai Linggunag Technique Co. Ltd., China) with 1.0 cm cells. Unless otherwise stated, all of the chemicals used were of analytical-reagent grade and all solutions were prepared with distilled water.

Ba²⁺ working solution (10 µg/mL): The standard stock solution of Ba²⁺ was prepared by placing 0.1437 g of BaCO₃ in 10 mL of hydrogen chloride (1:1, v/v) and by heating it for dissolution until after CO₂ was removed. After the content was cooled down, it was diluted to 100 mL with water that was cooked and boiled, shaken to obtain 1 mg/mL of Ba²⁺ stock solution. The working solution, 10 µg/mL, was prepared by dilution.

***p*-Acetylarsono solution:** 0.1 % (w/v) aqueous solution of *p*-acetylarsono was prepared. An NH₃-NH₄Cl buffer solution (pH = 10.4) was used.

General procedure: Place a definite amount (for conditional experiment: 30 µg) of Ba²⁺ in a 10 mL of calibrated flask and add 1.2 mL of NH₃-NH₄Cl buffer solution and 1.0 mL of chromogenic agent ASApA solution. Dilute to the mark with water and mix well. Measure the absorbance at 644 nm on a spectrophotometer in a 1.0 cm cell against a reagent blank.

Absorption spectra: Under the experimental conditions, ASApA and Ba(II) form a blue complex and its maximum absorption is 644 nm, while the absorption peak of ASApA reagent against water is located at 580 nm (Fig. 2). 644 nm was selected as the measurement wavelength in this study.

TABLE-1
ANALYTICAL RESULTS OF LAKE WATER

Sample	Found ($\mu\text{g/mL}$)	Average ($\mu\text{g/mL}$)	RSD (%)	Added ($\mu\text{g/mL}$)	Recovered ($\mu\text{g/mL}$)	Recovery (%)	DBS-Arsenazo spectrophotometric contrast method ⁶ ($\mu\text{g/mL}$)
Lake water	61.06, 64.64, 65.44, 60.26, 62.65, 65.44, 66.24, 61.46, 62.25, 63.05, 64.25, 61.85, 67.44	63.54	2.94	2.75	2.88	104.8	63.54

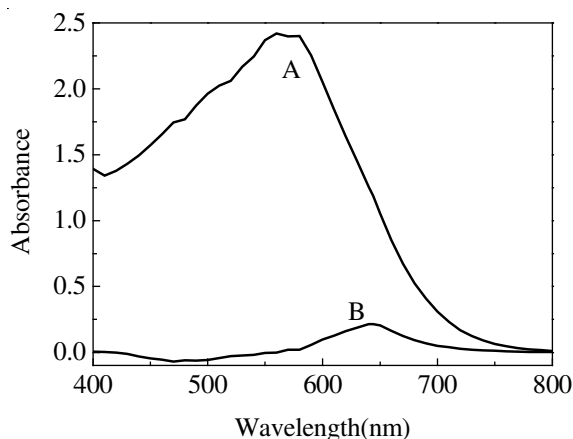


Fig. 2. Absorption spectra: (A) reagent blank against water; (B) complex against reagent blank. $[\text{Ba}^{2+}] = 2.18 \times 10^{-3} \text{ mol/L}$; $[\text{ASApA}] = 1.44 \times 10^{-4} \text{ mol/L}$; pH 10.4

Optimization of experimental conditions

Effect of acidity: It was experimentally found that when pH was in the range of 9.4-10.9, the absorbance was maximum. Therefore, $\text{NH}_3\text{-NH}_4\text{Cl}$ of pH 10.4 was chosen as the reaction medium. The experiments showed that when addition amount of the pH 10.4 $\text{NH}_3\text{-NH}_4\text{Cl}$ buffer solution was among 1.0-1.5 mL, the absorbance was the largest and stable. In the experiment, 1.2 mL was added.

Effect of the amount of chromogenic agent: Under the experimental conditions, different amount of 0.1 % (w/v) ASApA solution was added and absorbance was determined according to the general procedure. The results showed that when the amount of ASApA was 0.8-1.2 mL, the absorbance was the largest and stable. In the experiment, 1.0 mL was added.

Effect of the order of reagent addition: The experimental results showed that the order of reagent addition has not effect on the absorbance results determined. The order employed in this experiment was: $\text{Ba}^{2+} + \text{NH}_3\text{-NH}_4\text{Cl} + \text{ASApA}$.

Complex composition and stability: The composition was evaluated by the molar ratio method and continuous variation method. Both showed that the molar ratio of barium(II) to ASApA is 1:2.

Under the optimum experimental conditions, for the determination of $3.0 \mu\text{g/mL}$ Ba^{2+} , the results showed that under a room temperature speed of the colour reaction was very fast and absorbance of the colour reaction could instantly reach maximum and invariable and the formed complex could be stable for 5 h at least.

Analytical characteristics: The calibration graph was constructed according to the general procedure. The results showed that Beer's law was obeyed over the range 5-60 μg of

barium(II) in 10 mL of solution (0.50-6.0 $\mu\text{g/mL}$). Its regression equation was: $A = 0.063C$ (C : $\mu\text{g/mL}$)-0.0289, with a correlation coefficient of $\gamma = 0.9938$. The apparent molar absorptivity was calculated to be $\epsilon_{644 \text{ nm}} = 9.84 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$.

Eleven replicate analyses of a test solution containing 3.0 $\mu\text{g/mL}$ of Ba^{2+} by the general procedure gave a relative standard deviation of 1.37 %, indicating that the precision of the method was good and the reproducibility of the determination experiments was excellent. For eleven determination of a blank reagent, a standard deviation obtained was 0.36 % and the detection limit 3S/K (S is standard deviation and K is slope of the working curve) was calculated to be 0.17 $\mu\text{g/mL}$.

Effect of foreign ions: Solution containing 30 μg of Ba(II) and various amounts of foreign ions were prepared and determined by the general procedure. The tolerated limits (± 5 % error maximum) are shown as follows (μg): Na^+ (3000), Zn^{2+} (600), Sn^{2+} (600), Mn^{2+} (600), Cd^{2+} (600), Co^{2+} (90), Ni^{2+} (90), Mg^{2+} (60), Ni^{2+} (60), Cu^{2+} (6), Ca^{2+} (6), Pb^{2+} (3), B^{3+} (3000), Fe^{3+} (60), Cr^{3+} (15), Al^{3+} (6), La^{3+} (6), Bi^{3+} (3), Ti^{4+} (3), Ac^- (6000), NO_3^- (3000), Cl^- (3000), F^- (2400), SiO_3^{2-} (600), WO_4^{2-} (300), MoO_4^{2-} (300), SO_4^{2-} (100), PO_4^{3-} (300).

Analysis of sample: 50 mL of lake water was taken and 2 mL of aqua regia was added to it. The contents were evaporated on an electric cooker to near dryness, transferred to a 100 mL calibrated flask and diluted to the mark by water. 1 mL of the above sample was taken and determined according to the general procedure for barium content. Meanwhile, DBS-arsenazo photometry was used as contrast method⁶ for the determination of barium content in the water sample. The above results are present in Table-1.

Conclusion

This paper established a new method for determination of barium(II) using ASApA as chromogenic agent and successfully determined the barium content in the water sample. At 644 nm, Beer's law was obeyed over the range of 0.50-6.0 $\mu\text{g/mL}$ for Ba(II) and the regression equation of working curve was: $A = 0.063C$ (C : $\mu\text{g/mL}$)-0.0289. The detection limit of the method was 0.17 $\mu\text{g/mL}$.

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