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NOTE

Spectrophotometric Determination of Aluminum with Dibromo-*p*-sulfonic Acid Arsenazo

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The colour reaction between aluminum(III) and dibromo-*p*-sulfonic acid arsenazo takes place to produce a purple-red complex in the buffer solution of NH_4OAc-HOAc at pH 6.0. The maximum absorption wavelength of the complex is 610 nm. A good linearity is presented between absorbance and the content of Al(III) over the range of 0-1.4 μg/mL. Its linear regression equation is: A = 0.6930 C (C: μg/mL) + 0.0211, with a correlation coefficient of γ = 0.9992. The detection limit of the method is 12.2 ng/mL. The present method has been successfully applied to determine aluminum in water sample, with a relative standard deviation of 2.58 % for 10 determinations. The recovery of the method was 103.1 %.

Key Words: Aluminum, Dibromo-*p*-sulfonic acid arsenazo, Spectrophotometry.

Studies have shown that excess aluminum can interfere with the metabolism of phosphorus. It has an inhibitive effect on the activity of pepsin, leading to Alzheimer's disease, down syndrome and other diseases[1]. Aluminum has damage on the heart, liver, kidney and immune functions. Also, it has such embryo toxicity and teratogenicity[2]. Therefore, people established a variety of methods for determination of aluminum[3]. Spectrophotometry has the characteristics of low instrumentation price, operation simplicity and low determination cost. However, most of the spectrophotometric methods of aluminum have poor stability, great impact by medium acidity and many other shortcomings[3-7]. Therefore, studies of a simple and practical spectrophotometric method for the determination of aluminum is necessary and has a practical significance.

Dibromo-*p*-sulfonic acid arsenazo (DBS-ASA) (Fig. 1) has good stability. No report about colour reaction of the reagent with aluminum is seen.

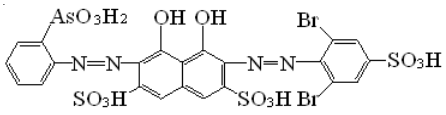

Fig. 1. Structure of DBS-ASA

The research discovers that the colour reaction between aluminum(III) and dibromo-*p*-sulfonic acid arsenazo takes place to produce a purple-red complex in the buffer solution of NH_4OAc-HOAc. Based on this, a new method for the spectrophotometric determination of aluminum was studied.

722S spectrophotometer (Shanghai Lingguang Technology Limited Corporation, China) was used for absorbance measurement using 1 cm cells.

Standard Al(III) stock solution (1.0 mg/mL): 1.7582 g $KAl(SO_4)_2 \cdot 12H_2O$ (G.R) was dissolved in 100 mL of water. It was diluted to 10 μg/mL working solution before use. DBS-ASA solution (Shanghai Changke Reearch Institute of Reagents, Jingsheng Chemical Industry Corp. Ltd., China): 0.1 g of DBS-ASA was dissolved in 100 mL of water. Buffer solution of NH_4OAc-HOAc at pH 6.0: 60 g NH_4OAc and 2 mL glacial HOAc were dissolved in water and diluted to 100 mL.

All the reagents used in the experiment were of analytical pure. Distilled water was used throughout the experiment.

Procedure: In 10 mL colorimetric tubes, 1 mL of 10 μg/mL Al(III) working solution, 1.7 mL of 0.1 % DBS-ASA, 1.5 mL of NH_4OAc-HOAc at pH 6.0 was added in turn. The resulting solution was diluted to the mark and shaken up. After 5 min, the absorbance of coloured solution was determined in 1 cm cells at 610 nm using the corresponding reagent blank as reference.

Absorption spectra: The colour reaction between aluminum(III) and dibromo-*p*-sulfonic acid arsenazo took place to produce a purple-red complex in the buffer solution of NH_4OAc-HOAc at pH 6.0. The absorption curve is shown

TABLE-1
ANALYTICAL RESULTS OF SAMPLE

Sample	Found (ng/mL)	Average (ng/mL)	RSD (%)	Added (μg/mL)	Recovered (μg/mL)	Recovey (%)	AAS method (ng/mL)
Lake water	27.3, 26.6, 26.4, 26.6, 27.8, 25.6, 27.6, 26.9, 28.0, 27.3	26.99	2.58	1.00	1.03	103.1	26.97

as Fig. 2. The maximum absorption of reagent blank *versus* water is at 510 nm. For the complex *vs.* reagent blank, the maximum absorption is at 610 nm. Thus, 610 nm was chosen as the measurement wavelength.

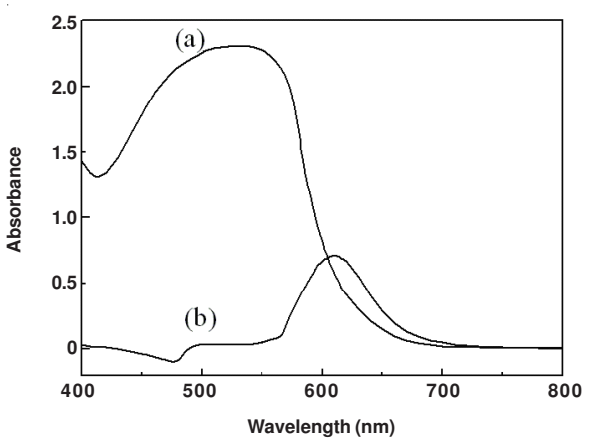

Fig. 2. Absorption spectra: (a) DBS-ASA (*versus* water); (b) complex (*versus* reagent blank); $[Al^{3+}] = 3.7 \times 10^{-5}$ mol/L; [DBS-ASA] = 2.0 $\times 10^{-4}$ mol/L; pH = 6

Effect of acidity: The effect of different acidity on the colour reaction of aluminum(III) and DBS-ASA was tested. The results showed that absorbance increased with pH between 3.5-5.8. Smooth change took place between 5.8-6.2 and the absorbance was maximum. At pH > 6.2, absorbance reduced. Thus, the buffer solution of NH_4OAc-HOAc at pH 6.0 was chosen. The results of the amount of buffer solution showed that when the dosage was 1.0-2.0 mL, the absorbance changed little. At 1.5 mL, absorbance was maximum. In the present experiment, 1.5 mL of buffer solution was selected.

Effect of DBS-ASA dosage: The effect of DBS-ASA solution with different volume was tested. The results showed that absorbance increased with increasing DBS-ASA dosage between 0-1.5 mL. Absorbance increased slowly over the range of 1.5-1.8 mL of DBS-ASA solution. Afterwards, as DBS-ASA dosage increased, the absorbance decreased. In this work, 1.7 mL of DBS-ASA solution was selected.

Influence of the adding sequence of reagent: The results showed that the addition order of reagent had no influence on the absorbance. In this paper, the addition order was as follows: Al^{3+} + DBS-ASA + buffer solution.

System stability: Under the optimum experimental conditions, the absorbance of 1.0 μg/mL Al^{3+} was determined at different time. After 5 min, the complex was found. The absorbance change was less than 5 % within 1.5 h.

Composition of complex: Under the optimum conditions, aluminum(III) and DBS-ASA produced a purple-red complex. Equimolar ratio method and the continuous variation method were applied to measure the composition ratio of the complex and obtained to be Al(III):(DBS-ASA) = 1:1.

Working curve: A standard curve was prepared under optimal conditions. The results showed that a good linear relationship lies in the range of 0-1.4 μg/mL. The regression equation was A = 0.6930 C (C: μg/mL) + 0.0211, with a correlation coefficient of 0.9992. The molar absorption coefficient was calculated to be $\varepsilon_{610\ nm} = 1.91 \times 10^4$ L mol^{-1} cm^{-1}. The eleven parallel determinations of 1.0 μg/mL aluminum(III) were made and the relative standard deviation obtained was 1.24 %. The blank test was repeated 11 times and the standard deviation was obtained. According to 3S/K method (S is the standard deviation of 11 reagent blank, K is the slope of working curve), the detection limit calculated is 12.2 ng/mL.

Influence of coexisting ions: Under the optimal conditions, for the determination of 10 μg aluminum(III) in a 10-mL solution the tolerant amount of coexisting ions was defined as the amount resulting in the error of ≤ ± 5 % and shown as follows (in μg): NH_4^+ (1000); Zn^{2+} (10000); Co^{2+} (800), Ni^{2+} (300); Mn^{2+}, Pb^{2+}, Ca^{2+}, Sr^{2+}, Mg^{2+} (100); Cu^{2+} (20); Bi^{3+}, Fe^{3+} (20); Cr^{3+} (5); La^{3+}, Ce^{3+}, Eu^{3+} (2); Si^{4+} (2000); Ti^{4+} (5); Th^{4+} (2); Cl^- (20000); NO_3^- (8000); Br^- (400); VO_3^-, NO_2 (100); MnO_4^- (5); $Cr_2O_7^{2-}$ (100); MoO_4^{2-} (20); PO_4^{3-} (50); ascorbic acid (500); oxalic acid (50); EDTA (50). For the interference of Fe^{3+}, a certain amount of phosphoric acid can be added to make it produce colourless complex. The impact of Cu^{2+} can be eliminated by thiourea.

Analysis of sample: 500 mL of lake water sample was taken and 5 mL of concentrated phosphoric acid was added for digestion to about 40 mL. Then, the content was filtered, transferred into a 50 mL volumetric flask. The filtrate was diluted with water to the mark. 5 mL of the testing solution was taken and determined according to the standard procedure. The results are seen in Table-1.

This study shows that the optimum conditions of the colour reaction between aluminum(III) and dibromo-*p*-sulfonic acid arsenazo. At 610 nm the good linearity is presented between absorbance and the content of Al(III) over the range of 0-1.4 μg/mL. Its linear regression equation is: A = 0.6930 C (C: μg/mL) + 0.0211, with a correlation coefficient of γ = 0.9992. The detection limit of the method is 12.2 ng/mL.

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