



EXTRACTION AND SPECTROPHOTOMETRIC DETERMINATION OF V (V) METAL IONS USING 5-BROMO SALICYLIDENE-2-AMINOTHIOPHENOL (BSATP) AS AN ANALYTICAL REAGENT

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ABSTRACT

A spectrophotometric method has been developed for the determination of V (V) using 5-bromo salicylidene-2-aminothiophenol¹⁻³ as an extractive reagent. The reagent forms a colored complex, which has been quantitatively extracted into chloroform at pH 4.8. The method obeys Beer's law over a range of 1 to 10 ppm. The molar absorptivity is $6084.0 \text{ L mol}^{-1} \text{ cm}^{-1}$ and Sandell's sensitivity is $0.0192 \mu\text{g cm}^{-2}$, respectively. The proposed method is very sensitive and selective. This method has been successfully applied to synthetic and commercial samples.

Key words: Vanadium, Spectrophotometric determination, Chloroform, 5-Bromo salicylidene-2-aminothiophenol.

INTRODUCTION

The cursory look at the literature survey reveals the fact that vanadium reacts with many organic reagents. It also indicates that some of the reagents recommended suffering through limitations such as complex formation takes place after several minutes⁴; some of the reagents are not selective⁵; sensitive also and some are less stable⁶. In this paper, a new method has been developed using 5-bromosalicylidene-2-aminothiophenol [BSATP] for extraction and spectrophotometric determination of vanadium, V (V), which is simple, selective and sensitive.

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EXPERIMENTAL

The reagent 5-bromosalicylidene-2-aminothiophenol was synthesized by the given procedure. The stock solution of V (V) was prepared by dissolving a weighed amount of ammonium metavanadate in double distilled water and then diluted to the desired volume with double distilled water and standardized by silver nitrate method⁷. The absorbance and pH measurements were carried out on a Shimadzu UV-Visible 2100 spectrophotometer with 1 cm quartz cells and digital pH meter with combined glass electrode, respectively.

Procedure for the extraction

0.1 mL of aqueous solution containing 1 μg of vanadium metal and 2 mL of reagent was mixed in a 50 mL beaker. The pH of the solution was adjusted to 4.8. It must be noted that the total volume should not exceed 10 mL. The solution was transferred to 100 mL separatory funnel. The beaker was washed twice with chloroform and transferred to the same funnel. The two phases were shaken for two minutes and allowed to separate. The organic phase was passed through anhydrous sodium sulphate in order to absorb trace amount of water from organic phase and then collected in 10 mL measuring flask and made up to the mark with organic solvent if required. The amount of vanadium present in the organic phase was determined quantitatively by spectrophotometric method by taking absorbance at 405 nm and that in the aqueous phase was determined by silver nitrate method.

RESULTS AND DISCUSSION

Extraction as a function of pH

The extraction of vanadium with 5-bromosalicylidene-2-aminothiophenol has been studied over the pH range 1-10 and it was observed that percentage extraction of V (V) is maximum at pH 4.8.

Absorption spectrum

The absorption spectrum of V (V)- 5-Bromosalicylidene-2-aminothiophenol in chloroform shows the maximum absorption at 405 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence, the absorption measurements were carried out at 380 nm.

Influence of diluents

The suitability of solvent was investigated using various organic solvents and the extraction of V (V). BSATP was quantitative in chloroform. Hence, chloroform was used for further extraction studies as it gave better and quicker phase separation.

Effect of reagent concentration

It was found that 2 mL of 0.1% reagent is sufficient for the colour development of the metal V (V) in 10 mL of aqueous solution at pH 4.8.

Effect of equilibration time and stability of the complex

The equilibration time of 1 minute is sufficient for the quantitative extraction of vanadium. The stability of colour of the V (V)- BSATP complex with respect to time shows that the absorbance due to extracted species is stable up to 48 hrs., after which slight decrease in absorbance is observed.

Calibration plot

The Beer's law is obeyed from 1 to 10 ppm. The molar absorptivity and sandell's sensitivity were calculated to be $6084.0 \text{ L mol}^{-1}\text{cm}^{-1}$ and $0.0192 \mu\text{g cm}^{-2}$, respectively (Fig 1).

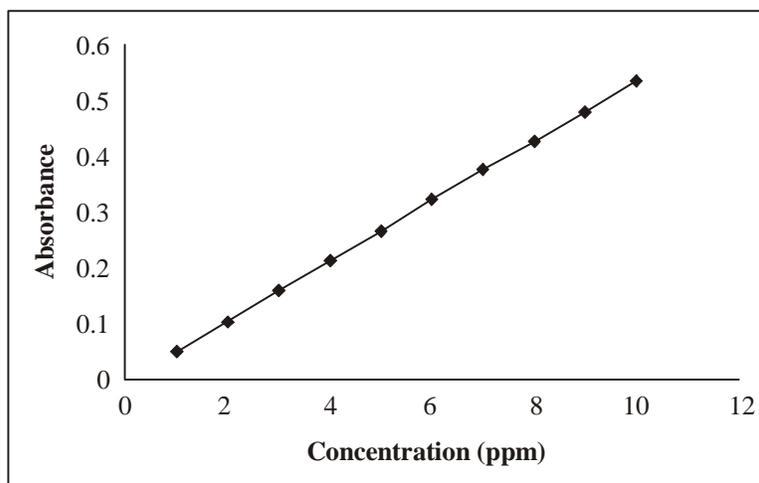


Fig. 1: Calibration plot of V (V)- BSATP complex

Effect of divalent ions and foreign ions

The effect of other ions present in various amounts indicated that there is no interference in the spectrophotometric determination of 10 ppm of vanadium. The ions, which show interference in the spectrophotometric determination of vanadium were overcome by using appropriate masking agents (Table 1).

Table 1: Effect of divalent ions and foreign ions

Interfering ions	Masking agents added
Se ⁴⁺	Oxidation with KMnO ₄
Th ⁴⁺	Sodium fluoride
Citrate, Tartarate	Sodium molybdate
Ag ⁺	Potassium iodide
Be ²⁺	Sodium fluoride
Fe ³⁺	Alkali cyanide/Thiourea
Cu ²⁺	EDTA/Sodium thiosulphate
Cr ³⁺	Ammonium acetate

Precision and accuracy

The precision and accuracy of the developed spectrophotometric method have been studied by analyzing ten solutions each containing 10 µg of vanadium in the aqueous phase. The average of ten determinations was 9.957 and variation from mean at 95% confidence limit was ± 0.1590.

Nature of extracted species

The composition of extracted V (V)- BSATP complex has been determined by Job's continuous variation methods, i.e. Slope ratio method and mole ratio method. It shows that the composition of V (V) - BSATP complex is 1 : 2 (Fig. 2).

Applications

The proposed method was successfully applied for the determination of vanadium

from various alloys, ores and pharmaceutical samples. The results were found to be in good agreement with those obtained by the standard known method (Table 2).

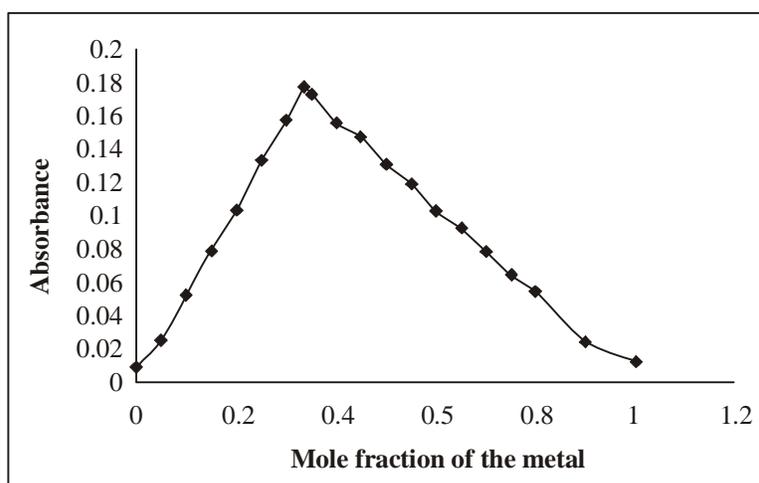


Fig. 2: Job's continuous variation curve

Table 2: Applications in different samples

Synthetic mixtures

Composition of sample (mg)	Vanadium added (mg)	Vanadium found (mg) by present method
Zn (4), W (6), Co (8)	8	7.90
Zr (5), Ti (2)	4	3.88
Cr (2), Mn (4), Pt (4)	5	4.93
Sr (3), Pb (2), Ru (4)	3	2.85

Steel alloys sample

Alloy	Certified value (%)	Observed value by present method (%)
Carbon steel	0.06	0.058
High speed steel super rapid extra 500	1	0.96

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