

New Analytical Technique for Determination of Trace Amount of V(V) by using UV-Visible Spectrophotometric Method with Photometric Reagent

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INTRODUCTION

Vanadium forms a light yellow colour complex with 2H3MBO at pH 2.0 which can be extracted quantitatively using n-butanol as an extractant. The absorbance was found maximum for 1 cm³ of 0.1% of methanolic solution of 2H3MBO. The absorption spectrum of V (V): 2H3MBO in n-butanol shows maximum absorption at λ_{max} 420 nm. At this wavelength Beer-Lambert's law is obeyed over the range of 1 to 10 μ g. Molar absorptivity and Sandell's sensitivity of the complex were calculated and found to be $6.0 \times 10^2 \text{ L mol}^{-1} \text{ cm}^{-1}$ and 0.0425 $\mu\text{g cm}^{-2}$ respectively. The composition of extracted V (V): 2H3MBO complex had been studied by Job's continuous variation method, mole ratio method and slope ratio method. On the basis of the results of these methods it can be concluded that the metal: ligand ratio is 1:2. The results of the prescribed procedure applied for the determination of the microamounts of V(V) in standard steel samples, alloys, pharmaceutical and synthetic samples are presented. The method was successfully applied to various pharmaceutical samples, alloys, ores and synthetic mixtures. The results were found to be in good agreement with the earlier known method.

EXPERIMENTAL

The pH measurements were made using a pH meter Elico, Model LI-129, India in conjugation with a combined glass and calomel electrode. Shimadzu UV-Visible 2100 spectrophotometer with 1.0 cm matched quartz cells were used for all absorbance measurements.

REAGENT AND CHEMICALS

0.1% 2H3MBO reagent is prepared by dissolving the requisite amount of 2H3MBO in a known volume of methanol. All chemicals used were of analytical-reagent grade or the highest purity available. Doubly distilled de-ionized water and A.R. grade methanol, which is were used throughout.

VANADIUM (V) SOLUTION

A weighed quantity of Ammonium metavanadate [NH_4VO_3] was dissolved in double distilled water containing dilute sulphuric acid and then diluted to desired volume using double distilled water. The Vanadium solution was standardized by Phosphotungstate method.

PROCEDURE FOR EXTRACTION

1cm³ of aqueous solution containing 100 μg of vanadium and 1cm³ of 0.1% solution of the reagent were used in a 50 mL beaker. The pH of the solution adjusted to 4.0, it must be noted that the total volume should not exceed 10mL. The solution was transferred to 100 mL separatory funnel. The beaker was washed twice with n- Butanol and transferred to the same

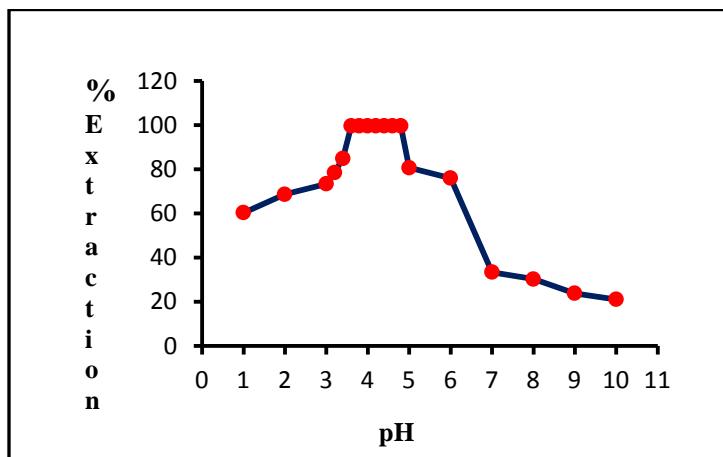
funnel. The two phases were shaken for two minutes and allowed to separate. The organic phase was passed through anhydrous sodium sulphate in order to absorb trace amount of water from organic phase and then collected in 10 mL measuring flask and made up to the mark with organic solvent if required. The amount of Vanadium present in the organic phase determined quantitatively by spectrophotometric method by taking absorbance at 420 nm and that in the aqueous phase was determined by Phosphotungstate method.

RESULTS AND DISCUSSION

The results of various studies are discussed below.

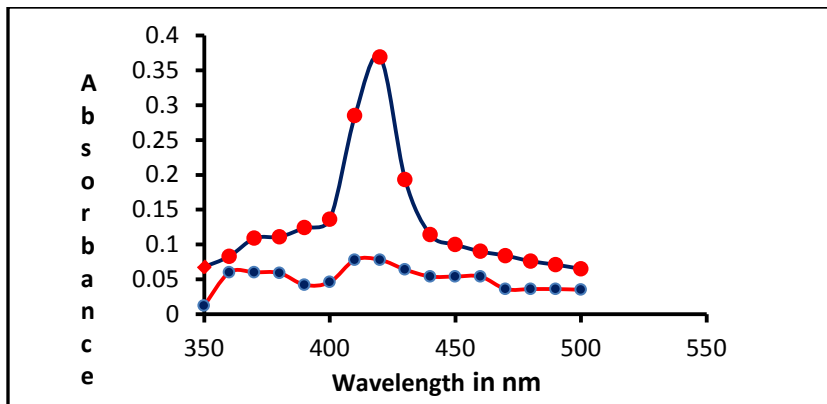
pH- Study

The extraction of vanadium with 2H3MBO was carried out over the pH range of 1 to 10 1cm^3 of aqueous solution containing $100\ \mu\text{g}$ of vanadium and 1cm^3 of 0.1% solution of the reagent were used. It reveals that 99.00 % and above of the metal is extracted into organic phase in the pH range 3.0 to 5.0 so the analytical work for the estimation of vanadium is carried out at pH 4.0. (Fig. 1).



Absorbance maxima

The absorption spectrum of V(V):2H3MBO complex in n-butanol shows maximum absorbance at $\lambda_{\text{max}} = 420\text{nm}$. The absorbance due to the reagent at this wavelength was negligible. Hence the wavelength 420nm was selected for further spectrophotometric study of V(V): 2H3MBO complex against the reagent blank. (Fig. 2)



Influence of diluents

The suitability of solvent was investigated using various organic solvents and the extraction of V(V): 2H3MBO was quantitative in n-butanol. Hence, n-butanol was used for further extraction studies as it gave better and quick phase separation.

Effect of reagent concentration

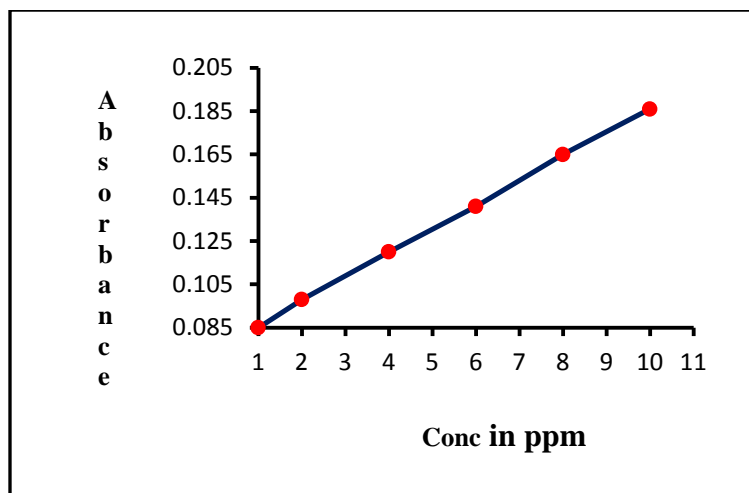
It was found that 1.0 mL of 0.1% reagent is sufficient for the colour development of the metal V(V) in 10 mL of aqueous solution at pH 4.0.

Effect of equilibration time and stability of the complex

The equilibration time of 1.0 minute is sufficient for the quantitative extraction of Vanadium. The stability of color of the V(V): 2H3MBO complex with respect to time shows that the absorbance due to extracted species is stable up to 36 hours, after which slight decrease in absorbance is observed.

Calibration plot

A linear plot was obtained when the measured absorbance values are plotted against the amount of V(V) in the concentration range of $1\mu\text{g}/\text{cm}^3$ to $10\mu\text{g}/\text{cm}^3$ at 420nm. The Molar Absorptivity and Sandell's sensitivity were calculated and found to be $6.0 \times 10^2 \text{ Lmol}^{-1}\text{cm}^{-1}$ and is 0.0425cm^{-2} .



Limit of detection

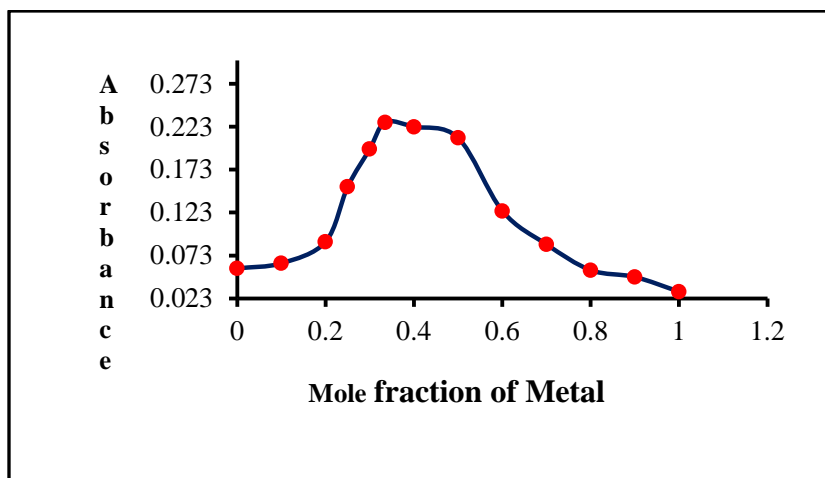
1 cm³ of 0.1 % methanolic 2H3MBO is diluted with buffer of pH 4.0 which is then extracted with n-butanol as per the procedure described earlier and then its absorbance is measured at 420 nm by taking solvent as a blank. The process is repeated for five times separately and five observations are obtained. The limit of detection for Vanadium for the method was found to be 0.296 ppm.

Precision and accuracy

The precision and accuracy of the spectrophotometric method were tested by analyzing the solution containing known amount of vanadium. Average of ten determinations of 6 μg V (V) in 10 cm.³ solution is 5.980 μg which varies between 5.980 μg to 6.014 μg at 95% confidence limit.

Nature of extracted species

The composition of extracted V(V): 2H3MBO complex has been determined by Job's continuous variation method, Slope ratio method and Mole ratio method. It shows that the composition of V(V): 2H3MBO complex is 1:2. (Fig 4).



Application The proposed method was successfully applied for the determination of Vanadium from various alloys, pharmaceutical and synthetic samples. The results found to be in good agreement with those obtained by the standard known method.

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